

# The American Mineralogist

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Society of America*

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Volume 5, Part 1

Sponsored by The Walker Mineralogical Club

Edited by M. A. Peacock, Toronto

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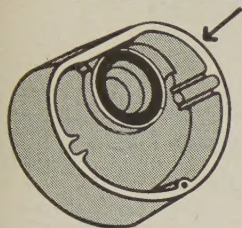
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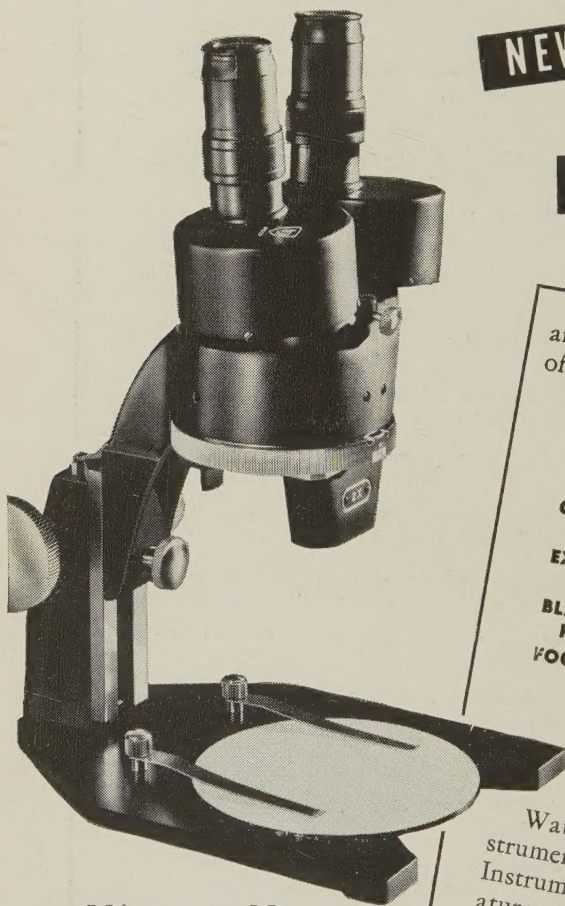


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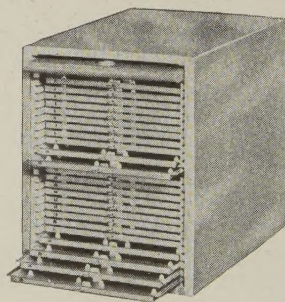
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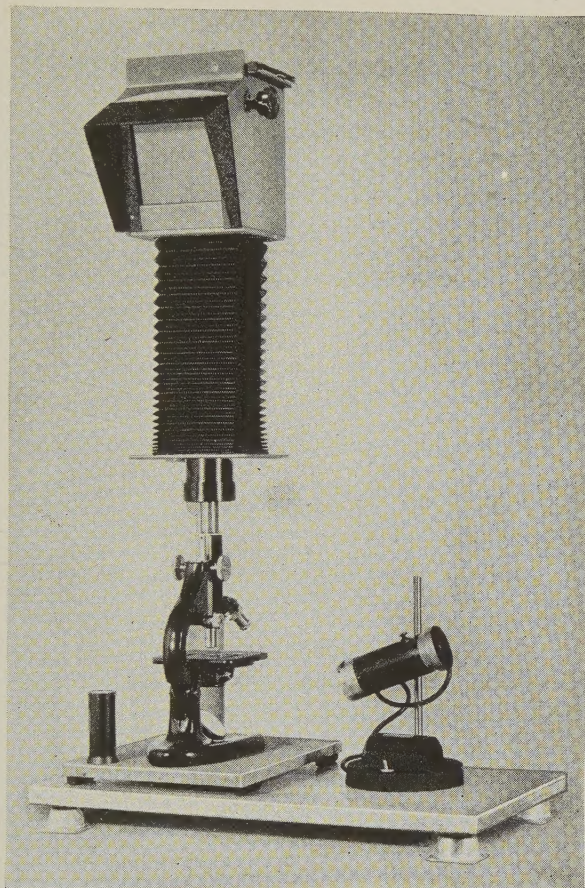
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## Contributions to Canadian Mineralogy

Volume 5, Part 1

Sponsored by **The Walker Mineralogical Club**

Edited by **M. A. Peacock, Toronto**

### FOREWORD

In 1921, shortly after the organization of the Mineralogical Society of America (December 30, 1919), Professor T. L. Walker (1867-1942) and Professor A. L. Parsons (now emeritus)—both charter fellows and in later years presidents of the Society—founded *Contributions to Canadian Mineralogy* from the Department of Mineralogy and Petrography, University of Toronto. This annual publication was conducted by Professors Walker and Parsons and, since 1941, by Professor M. A. Peacock; and from 1921 to 1948 it has appeared without interruption in the *Geological Series* of the *University of Toronto Studies*. Since the founding of the Walker Mineralogical Club in 1938, the periodical has been sponsored by the Club as well as by the University; and after the union of the Department of Mineralogy and Petrography and the Department of Geology and Palaeontology in 1945, the Canadian journal was issued by the combined Department of Geological Sciences and the Walker Mineralogical Club.

At the November 1948 meeting of the Council of the Mineralogical Society of America, Professor (then President) Peacock reported that the University of Toronto had decided to terminate the *University of Toronto Studies* which, as a whole, had greatly declined, and that no practical way had been found to continue the publication of *Contributions to Canadian Mineralogy* in Canada. In order to preserve the continuity and integrity of the Canadian Journal, the Council agreed to devote a regular issue of the *American Mineralogist* to a collection of papers by Canadian mineralogists, to be assembled and edited by Professor Peacock in consultation with the Editor of the *American Mineralogist*. With this first issue of *Contributions to Canadian Mineralogy* as a part of *The American Mineralogist*, it is a pleasure to assist our Canadian colleagues to publish and distribute their valuable mineralogical results.

WALTER F. HUNT



# THE TELLURIDE MINERALS AND THEIR OCCURRENCE IN CANADA<sup>1</sup>

R. M. THOMPSON<sup>2</sup>

*University of British Columbia, Vancouver, B. C.*

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## ABSTRACT

A study of telluride minerals and ores, mainly by microscopic and  $x$ -ray methods, has led to revised and condensed synoptic descriptions of the telluride minerals and a catalogue of Canadian telluride localities. The mineral descriptions contain new measurements of specific gravity, some new cell dimensions, and numerous new  $x$ -ray powder patterns, given in tables and reproductions; they also include the available chemical analyses of Canadian materials and summaries of observed occurrences and associations. The topographic list of Canadian telluride localities distinguishes positive identifications by  $x$ -rays from those based on older methods.

## INTRODUCTION

The telluride minerals as a group are rarer than the sulphides and less well known, and consequently they are of special interest to the ore mineralogist. At the same time the tellurides are unusually interesting to the mining geologist and the prospector, as compounds of gold and silver or as close associates and indicators of the precious metals. The four main telluride districts are Nagyág (Transylvania), Kalgoorlie (Western Australia), Cripple Creek (Colorado), and Kirkland Lake (Ontario), and all of these are also important gold producing districts. The telluride min-

<sup>1</sup> Extracted from an unpublished Ph.D. thesis: Descriptive mineralogy of the tellurides—*University of Toronto*, 1946.

<sup>2</sup> Assistant Professor of Mineralogy, Department of Geology.



erals of the Transylvanian deposits have been described principally by Schrauf (1878), those of Kalgoorlie by Stillwell (1931), those of Cripple Creek by Genth (1868, 1877), Penfield & Ford (1902), and others, and the Canadian localities have been recorded by Cairnes (1912), Todd (1929), and especially Ellis Thomson (1922-1937).

Until recently the known Canadian telluride localities have been almost confined to Ontario and Quebec. The discovery of a number of bismuth tellurides in British Columbia by Dr. H. V. Warren led to an intensive study of these minerals and the bismuth-tellurium system by Dr. M. A. Peacock (1941; Warren & Peacock, 1945), and as a result the members of this group and their relations are better known. As a student and assistant of Professor Warren in Vancouver and later of Professor Peacock in Toronto, the present writer became interested in the occurrence of tellurides in British Columbia and their persistent association with gold, and this led him to an extended study of these minerals and their occurrence in Canada.

#### METHODS OF STUDY

The study of a species commenced with an examination of hand specimens in daylight, and under the binocular microscope. This revealed such properties as cleavage, colour, tarnish, and lustre, and permitted the determination of hardness, tenacity, and mineral association. Numerous specific gravity measurements were made on selected fragments, with the Berman torsion microbalance, which uses samples of 10-20 mg. The minerals studied are all opaque and therefore polished sections were made to test the homogeneity of the material, to observe the optical properties in reflected light and the behaviour to the standard etch-reagents as recorded by Short (1940), and occasionally to determine paragenetic relationships. Numerous *x*-ray powder photographs were made to compare known and doubtful materials. Whenever the elements of the unit cell were known or could be determined, the powder pattern was indexed as far as it seemed reasonable, giving standard *x*-ray powder patterns with every line accounted for.

Telluride minerals often occur in small amount in ore, in intimate association with sulphides and gold. Precise determination is rarely possible in such small areas by the usual microscopic means, as many of the tellurides have similar properties, and numerous errors have been made in identification. In general, there is great need for corroborative evidence in the determination of the rarer minerals in polished sections. Such minerals are often named on the basis of optical behaviour, etch-tests, and micro-chemical tests which are not always sufficient. Mistakes in identification lead to embarrassment to the writers and confusion in the litera-

ture. It is hoped that the time is rapidly approaching when all but the commonest ore minerals will be positively identified by  $x$ -ray means.

The following procedure, based on the methods of Haycock and Harcourt, was used to determine minerals present in areas as small as 20 microns in diameter in a polished section. A needle with a very sharp point was applied to the area in question and a minute quantity detached by slight scraping. A drop of collodion was placed on the scraping and allowed to become almost dry before being collected by the needle and transferred to the point of a finger where it was rolled into a small ball by another finger. This ball was then placed on the end of a thin filament of glass and centered in the  $x$ -ray camera. The resulting powder pattern on a fraction of a milligram usually served definitely to identify the substance. In some cases, where the area scraped was less than 20 microns in diameter, the pattern of the adjacent mineral or minerals was also present and could often be identified.

#### SCOPE OF THE PRESENT PAPER

A full account of the writer's work on the telluride minerals is given in an unpublished Ph.D. thesis, and some of the results of this work have already been published (Thompson, 1946*a*, 1946*b*, 1947; Peacock & Thompson, 1946*a*, 1946*b*). The present paper is restricted to critically revised and condensed synoptic descriptions of all the known telluride minerals, together with a catalogue of occurrences in Canada. The descriptions emphasize the characters that can be determined by simpler means, and most of the information has been confirmed or improved by direct observation. Many new observations, notably measurements of specific gravity, cell dimensions and  $x$ -ray powder patterns, are included. To save space the indices ( $hkl$ ) and the calculated spacings of the powder patterns have been omitted. The catalogue of Canadian telluride occurrences and associations distinguishes those which have been established by  $x$ -ray photographs from those based on earlier determinations. An account of a study of the pyrosynthesis of the tellurides is reserved for another occasion, and all discussion of the origin and economic value of the tellurides is excluded from this purely descriptive paper. For convenience the tellurides are grouped according to the principal metal constituents: gold (montbrayite, calaverite, krennerite); gold and silver (petzite, sylvanite); silver (hessite, empressite); copper (weissite, rickardite); nickel (melonite); iron (frohbergite); lead (altaite, nagyagite); mercury (coloradoite); bismuth (hedleyite, joseite, tellurbismuth, wehr-lite, tetradymite); and finally the uncertain or discredited tellurides, stuetzite, muthmannite, niggliite, "antamokite" and "goldfieldite" are briefly mentioned.



## MATERIAL AND ACKNOWLEDGMENTS

In the examination of any mineral species authentic material is of great importance. I wish to express my thanks to Dr. V. B. Meen of the Royal Ontario Museum of Geology and Mineralogy who kindly placed at my disposal many excellent and typical specimens; to Mr. R. A. Bryce of Macassa Mines for several spectacular specimens of ore from the Robb-Montbray Mine, Montbray Township, Quebec; to Dr. M. H. Froberg for a large number of specimens from northern Ontario, Quebec and elsewhere; to Dr. H. Hopkins for specimens from the Wright-Hargreaves Mine, Kirkland Lake, Ontario; to Dr. E. P. Henderson of the United States National Museum for type specimens of *empresite* and "*antamokite*"; to Professor H. E. T. Haultain for specimens of *sylvanite* and native tellurium; to Mr. S. A. Pain for an excellent specimen of *hessite* and *petzite*; to the several mining companies in Ontario and Quebec for supplying specimens of telluride ores for study; to Dr. H. V. Warren of the University of British Columbia for several specimens of telluride ores. Finally I am much indebted to Professor M. A. Peacock, under whom this work was carried out, for spending much time assisting me in the course of this study and in the preparation of this report.

MINERAL DESCRIPTIONS<sup>3</sup>*Tellurides of Gold***Montbrayite**— $\text{Au}_2\text{Te}_3$ 

Triclinic,  $P\bar{1}$ ;  $a=12.08$ ,  $b=13.43$ ,  $c=10.78$ ,  $\alpha=104^\circ 30\frac{1}{2}'$ ,  $\beta=97^\circ 34\frac{1}{2}'$ ,  $\gamma=107^\circ 53\frac{1}{2}'$ ;  $Z=12$  (Peacock & Thompson, 1946b).

Massive but sometimes with interrupted parting planes. Yellowish-white colour, paler than *calaverite*, with splendid metallic lustre. Very brittle, breaking with a flat-conchoidal fracture;  $H=2\frac{1}{2}$  (C);  $G=9.94$ .

Polished sections creamy-white like *krennerite* and distinctly less white than *altaite*. Reflection pleochroism rarely perceptible; weak to moderate anisotropism (light gray, light yellow-brown, blue-gray). The mineral makes coarse solid mosaics with optically continuous areas often several millimeters in width.  $\text{HNO}_3$  (1:1), strong effervescence with a light yellow-brown stain; several small (50 micron) circular areas staining light gray; after the drop has been removed an "alligator-skin" etch-pattern develops leaving the circular areas in relief;  $\text{HNO}_3$  (3:2), slower effervescence; surface stains light brown with development of small circular areas similar to the above which are also stained light brown; after the drop has been removed an etch-pattern develops with one prominent direc-

<sup>3</sup> Lattice dimensions throughout this paper are in kX units. New measurements are based on the wavelengths  $\text{CuK}\alpha_1=1.5374$ ,  $\text{FeK}\alpha_1=1.9321$  kX.

tion; an epidermis a few microns thick tends to come off and curl up at the edges; this may be completely removed with hard buffing, the surface again becoming apparently homogeneous;  $\text{HNO}_3$  (conc.), very slow effervescence; surface stains uniformly light gray-brown; no etch-pattern develops after the drop has been removed; circular areas absent;  $\text{HCl}$ ,  $\text{KCN}$ ,  $\text{FeCl}_3$ ,  $\text{KOH}$ ,  $\text{HgCl}_2$ , negative.

*X-Ray Powder Pattern* (Peacock & Thompson, 1946b; fig. 1)

<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$
1	5.9	7.48	1	21.3	2.12	2	31.8	1.459
2	9.9	4.47	10	21.65	2.08	$\frac{1}{2}$	32.2	1.443
$\frac{1}{2}$	10.9	4.07	1	22.2	2.03	$\frac{1}{2}$	33.5	1.393
$\frac{1}{2}$	11.65	3.81	1	22.9	1.975	$\frac{1}{2}$	35.05	1.339
$\frac{1}{2}$	12.6	3.52	1	23.8	1.905	1	35.55	1.322
1	13.8	3.22	$\frac{1}{2}$	24.45	1.857	2	35.95	1.309
8	15.0	2.97	$\frac{1}{2}$	24.75	1.836	$\frac{1}{2}$	39.4	1.211
8	15.25	2.92	$\frac{1}{2}$	25.5	1.786	2	39.9	1.198
$\frac{1}{2}$	18.1	2.47	2	26.6	1.717	1	47.6	1.041
1	18.9	2.37	2	26.9	1.699	$\frac{1}{2}$	55.1	0.937
1	19.75	2.27	2	31.05	1.490	1	56.1	0.926

*Occurrence:* This species was found at the Robb-Montbray Mine, Montbray Township, Quebec, and described in detail by Peacock & Thompson (1946b). The gold telluride is abundant in some specimens (about 20 specimens are known to have been preserved) forming equidimensional masses sometimes exceeding 1 cm. in diameter. Freshly broken surfaces show coarsely crystallized aggregates of montbrayite, tellurbismuth, altaite, melonite, gold, chalcopyrite, and pyrite. In polished sections the following minerals are found in contact with montbrayite or enclosed within the gold telluride; altaite, tellurbismuth, petzite, gold, chalcopyrite, melonite, and frobergite. Altaite, with small amounts of gold and petzite, occurs in thin stringers cutting montbrayite. Tellurbismuth with subordinate altaite and small patches of petzite make ovoid inclusions with a eutectoid structure, irregularly distributed through montbrayite. In total bulk these inclusions might amount to 10 per cent of the montbrayite and in some cases they reach 1 mm. in diameter. In ordinary light the altaite shows as straight or curved bluish-white bands within the tellurbismuth, which is faintly reddish-white by contrast and not easily distinguished from the surrounding montbrayite. Crossed nicols emphasize the anisotropic tellurbismuth, and staining with  $\text{HCl}$  darkens the bands of altaite. These inclusions have the appearance of exsolution products. Petzite occurs as irregular areas often surrounding and traversing patches of montbrayite, and occasionally cutting altaite,



tellurbismuth, and melonite. Subhedral crystals of melonite, with or without partial rims of petzite, occur in montbrayite, but the best crystal outlines are shown by isolated plates of melonite in altaite. The chalcopyrite does not occur in the montbrayite itself but at the boundaries of altaite, tellurbismuth, petzite, and gold with montbrayite. Gold occurs abundantly in areas rich in pyrite and replaces subhedral crystals of the latter. Marcasite, chalcocite, and covellite are present in minor amounts.

<i>Analysis</i>	1	A
Au.....	44.32	50.77
Ag.....	0.55	—
Pb.....	1.61	—
Bi.....	2.81	—
Sb.....	0.90	—
Te.....	49.80	49.23
	<u>99.99</u>	<u>100.00</u>

1. Montbrayite with visible inclusions of tellurbismuth, altaite, and petzite. Anal. Williams (in Peacock & Thompson, 1946b).

A. Calculated for  $\text{Au}_3\text{Te}_3$ .

#### Calaverite— $\text{AuTe}_2$

Monoclinic,  $C2/m$ ;  $a=7.18$ ,  $b=4.40$ ,  $c=5.07$ ,  $\beta=90^\circ\pm 30'$ ;  $Z=2$  (Tunell & Ksanda, 1935).

Bladed or lath-like crystals, stout or slender prisms, also massive, granular to indistinctly crystalline. Pale brass-yellow in colour like pyrite, with high metallic lustre; twice observed with a peculiar bronzy-purple tarnish. Very brittle, breaking with a sub-conchoidal to uneven fracture; no cleavage.  $H=2\frac{1}{2}$ –3 (C);  $G=9.10$ –9.40 (R.M.T.).

Polished sections creamy-white with moderate anisotropism (light gray, brown, dark gray), showing moderately coarse-grained mosaics.  $\text{HNO}_3$  (1:1) stains light brown with effervescence; after the drop has been removed and the surface dried, an etch-cleavage develops showing one prominent direction; a few small circular areas similar to those developed in montbrayite and krennerite are less affected;  $\text{HNO}_3$  (3:2) stains light brown with effervescence; after the drop has been removed and the surface dried a two directional etch-pattern with several relatively unetched circular areas develops; with hard buffing the etch-cleavage pattern and circular areas may be removed, the surface underneath becoming perfectly smooth and apparently homogeneous;  $\text{HNO}_3$  (conc.) gives purplish-brown stain and a series of very fine closely spaced lines;  $\text{HCl}$ ,  $\text{KCN}$ , negative;  $\text{FeCl}_3$  slowly stains light brown;  $\text{KOH}$ ,  $\text{HgCl}_2$  negative. These observations on unoriented sections are in good agreement with those given by Short (1937).

*X-Ray Powder Pattern (R.M.T.; fig. 2)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
1	8.8	5.02	1	31.8	1.459	2	50.1	1.002
$\frac{1}{2}$	11.8	3.76	$\frac{1}{2}$	32.5	1.431	3	52.1	0.974
10	14.8	3.01	2	33.9	1.378	2	54.1	0.949
3	15.25	2.92	3	35.1	1.337	$\frac{1}{2}$	55.4	0.934
4	20.5	2.19	3	35.9	1.311	$\frac{1}{2}$	56.7	0.920
8	21.6	2.09	2	38.05	1.247	4	59.9	0.888
2	21.95	2.06	$\frac{1}{2}$	38.8	1.227	1	63.7	0.857
1	22.5	2.01	$\frac{1}{2}$	39.4	1.211	1	65.7	0.843
2	23.4	1.936	4	40.05	1.195	1	69.9	0.819
$\frac{1}{2}$	24.7	1.840	$\frac{1}{2}$	41.5	1.160	1	71.2	0.812
$\frac{1}{2}$	25.5	1.786	$\frac{1}{2}$	44.65	1.094	1	72.1	0.808
3	26.0	1.754	1	45.4	1.081	1	76.9	0.779
3	27.15	1.685	1	45.9	1.070	1	82.3	0.776
1	30.0	1.537	2	47.2	1.048			
3	30.8	1.501	2	48.4	1.028			

*Occurrences:* Rhodesia, Western Australia, Philippine Islands, Salvador, New Mexico, California, Colorado. At Kalgoorlie, Western Australia, as small compact masses ( $2 \times 5$  mm.) in a chip of chlorite schist; at the Benguet Consolidated Mine, Antamok, Mountain Province, Philippine Islands, in a specimen of somewhat vuggy quartz with disseminated chalcopyrite, gold, sphalerite, tetrahedrite, galena, calaverite, and pyrite; at Cripple Creek, Colorado, in several specimens of a fine-grained volcanic rock with fine-grained purple fluorite and calaverite as thin laths ( $1 \times 5$  mm.) or disseminated; also as two small crystals. Polished sections from Cripple Creek, Colorado, show calaverite as large irregular lath-like areas in gangue. The mineral contains small inclusions of pyrite and also replaces it; no free gold was observed in the sections.

In Canada calaverite has been identified by x-ray photographs at the Wright-Hargreaves, Bidgood, Macassa, Toburn, Lake Shore, Kirkland Golden Gate, and Upper Canada Mines, Kirkland Lake Area, Ontario, as small compact masses or disseminations in quartz, calcite, or altered porphyry, in association with other tellurides, sulphides, and free gold; at the Miller Independence Mine, Boston Creek, Pacaud Township, in a polished section with chalcopyrite, pyrite, and sphalerite. Also at the Lamaque Mine, Bourlamaque Township, Quebec, in a specimen of white quartz with small plates of tellurbismuth and massive calaverite; at the Louvicourt Goldfields Mine, Louvicourt Township, Quebec, in several specimens of tourmalinized quartz with much free gold and small areas of calaverite and tarnished tellurbismuth in close association with calcite; at the Canadian Malartic Gold Mine, Fourniere Township, Quebec, as



massive milky quartz with occasional 1 mm. grains of calaverite; and at the Horne Mine, Noranda, Quebec, in several pieces of greenstone with altaite, petzite, and calaverite in coarsely crystalline masses or intergrown with tellurbismuth.

Calaverite has also been reported from British Columbia, as a brass-yellow telluride (apparently calaverite) from the Engineer Mines, Taku Arm, Tagish Lake, Atlin Mining Division (Cairnes, 1912, p. 193); from Glacier Gulch, near Smithers (Pratt, 1931, p. 56); in Ontario from Painkiller Lake, Beatty Township (Hopkins, 1915, p. 180); the Boston McRae Mine, Pacaud Township (Thomson, 1922, p. 97); from Bigstone Bay, Lake of the Woods (Thomson, 1935, p. 48); from the Teck-Hughes and Sylvanite Mines, Kirkland Lake (Todd, 1929, pp. 81-83).

Calaverite from the Macassa Mine, Kirkland Lake, Ontario, occurs disseminated throughout quartz, also with gold, filling fractures in quartz. Large areas of calaverite often have small particles of gold attached. The calaverite appears to have replaced the fine grained pyrite with which it is associated. The writer's observations are in agreement with those of other workers as to the paragenesis at Cripple Creek, and Kirkland Lake, namely that the sulphides were formed first followed by tellurides and finally gold.

<i>Analyses</i>	1	2	A
Au.....	39.36	38.55	43.59
Ag.....	0.30	0.22	—
Pb.....	5.20	6.49	—
Cu.....	0.24	trace	—
Fe.....	0.33	0.70	—
Te.....	54.32	52.70	56.41
S.....	0.12	0.55	—
Insol.....	0.24	0.60	—
	<hr/> 100.11	<hr/> 99.81	<hr/> 100.00

1. Calaverite, Lake Shore Mine, Kirkland Lake, Ontario, with minor altaite; incl. Hg, Bi, Se, none. Anal. Rickaby (in Todd, 1929, p. 77).

2. Calaverite, Wright-Hargreaves Mine, Kirkland Lake, Ontario, with minor altaite, pyrite, and chalcopyrite; incl. Hg, Bi, Se, none. Anal. Rickaby (in Todd, 1929, p. 78).

A. Calculated for  $\text{AuTe}_2$ .

#### **Krennerite**— $(\text{Au}, \text{Ag})\text{Te}_2$

Orthorhombic,  $Pbm2$ ;  $a=8.80$ ,  $b=16.51$ ,  $c=4.45$ ;  $Z=8$  (Tunell & Ksanda, 1936).

Highly modified short vertically striated prismatic crystals, cleavage fragments, and grains. Silver-white in colour tarnishing to light brass-yellow. Perfect basal cleavage and uneven fracture. Brittle;  $H=2\frac{1}{2}$  (C);  $G=8.62$ .

Polished sections creamy-white, weakly anisotropic (light gray to dark gray) on a basal section.  $\text{HNO}_3$  (1:1) stains light brown with effervescence; after the drop has been removed, a distinct two directional etch-cleavage pattern develops;  $\text{HNO}_3$  (3:2) gives a similar stain and etch-cleavage pattern appears but relatively unetched circular areas appear irregularly distributed over the surface;  $\text{HNO}_3$  (conc.) stains light brown and develops a few irregular flakes;  $\text{HCl}$ ,  $\text{KCN}$  negative;  $\text{FeCl}_3$  stains light yellow;  $\text{KOH}$  gives light gray-brown tarnish;  $\text{HgCl}_2$  negative.

*X-Ray Powder Pattern (R.M.T.; fig. 3)*

<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$
1	9.4	4.71	3	21.8	2.07	3	31.7	1.463
3	11.25	3.94	1	22.95	1.971	2	33.35	1.398
10	14.60	3.05	1	23.55	1.924	3	34.75	1.348
4	15.05	2.96	3	25.65	1.776	4	35.8	1.314
1	19.15	2.34	$\frac{1}{2}$	26.25	1.738	1	37.45	1.264
4	20.1	2.24	3	27.0	1.693	1	38.75	1.228
5	21.35	2.11	3	30.45	1.517	3	39.85	1.199

*Occurrences:* Transylvania, Western Australia, Colorado. At Kalgoorlie, Western Australia, krennerite was identified as a small cleavage fragment ( $2 \times 2$  mm.) with a particle of attached petzite; and at Cripple Creek, Colorado, in a fine grained volcanic rock, disseminated, and as short striated prisms on one surface.

In Canada krennerite has been reported in Ontario, from the Ashley Mine, Bannockburn Township (Thomson, 1932, p. 27); from the McKenzie Red Lake Gold Mines, Red Lake (Hoiles in Horwood, 1945, p. 166) (the writer examined Hoiles' polished sections but observed only galena and tetradymite); and in Quebec, from the Robb-Montbray Mine, Montbray Township (Thomson, 1928, p. 12) (actually montbrayite), and from the Horne Mine, Noranda (Price, 1934, p. 132). There are no analyses of reported Canadian occurrences.

### *Tellurides of Gold and Silver*

#### **Petzite**— $\text{Ag}_3\text{AuTe}_2$

Crystal system unknown, usually massive with no trace of crystal form. Bright steel-gray with a mauve tinge to nearly jet-black, tarnishing from bronze-yellow to sooty black. No cleavage but slightly sectile to brittle with a sub-conchoidal fracture.  $H = 2\frac{1}{2}$  (A+);  $G = 9.13$  (R.M.T.).

Polished sections galena-white but violet-gray to dull gray in contact with other tellurides, sulphides, or gold; rarely shows triangular pits; isotropic.  $\text{HNO}_3$  quickly stains iridescent;  $\text{HCl}$  stains iridescent;  $\text{KCN}$



negative;  $\text{FeCl}_3$  quickly stains iridescent; KOH negative;  $\text{HgCl}_2$  slowly stains brown.

Petzite is not readily distinguished from coloradoite, empressite, or some types of tetrahedrite in hand specimens. Coloradoite, empressite, and tetrahedrite are quite brittle and show no sign of sectility when tested with a steel needle under a binocular. Petzite is at first slightly sectile but with increasing pressure particles snap off. The brownish-red streak of tetrahedrite helps to distinguish it from petzite. If a purple tarnish is not present on coloradoite, the mineral is indistinguishable from petzite.

*X-Ray Powder Pattern (R.M.T.; fig. 4)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
2	5.9	7.5	1	30.25	1.525	$\frac{1}{2}$	43.6	1.114
2	12.1	3.67	1	31.0	1.492	$\frac{1}{2}$	44.25	1.101
$\frac{1}{2}$	13.6	3.27	1	31.8	1.458	$\frac{1}{2}$	44.9	1.089
1	14.9	2.99	1	32.4	1.434	1	46.0	1.069
10	16.1	2.77	1	33.15	1.405	$\frac{1}{2}$	47.5	1.043
$\frac{1}{2}$	17.25	2.59	2	33.85	1.380	$\frac{1}{2}$	49.95	1.004
3	18.35	2.44	3	35.85	1.312	1	51.1	0.988
3	19.4	2.31	1	36.5	1.292	$\frac{1}{2}$	53.1	0.961
5	21.3	2.11	1	37.2	1.271	$\frac{1}{2}$	56.7	0.920
4	22.3	2.02	1	39.8	1.200	$\frac{1}{2}$	59.3	0.894
3	23.9	1.897	1	41.05	1.170	1	60.9	0.880
2	24.9	1.826	$\frac{1}{2}$	42.3	1.142	1	62.35	0.868
1	29.55	1.558	$\frac{1}{2}$	43.05	1.126			

*Occurrences:* Transylvania, Western Australia, Colorado; also reported from New Mexico, California, Oregon, New Zealand, Korea, Siberia, and Asia Minor. Identified at Kalgoorlie, Western Australia, as several small chips associated with coloradoite and krennerite; at Gold Hill, Boulder County, Colorado, as a compact mass  $10 \times 10$  mm.; and at the Red Cloud Mine, Boulder County, Colorado, as a compact mass associated with tetradymite and pyrite.

In Canada, the occurrence of petzite from numerous localities has been confirmed by *x*-ray powder photographs. In British Columbia, at the Hedley Monarch Mine, Osoyoos Mining Division, as minute areas in polished section associated with altaite and hessite. In Manitoba, at the San Antonio Mine, near Bissett, in a specimen of quartz with small areas of calcite containing abundant gold and small grains of petzite. In Ontario at the Ardeen (Moss) Mine, Moss Township, together with hessite, as a massive black crust 5 mm. thick on one surface of vein quartz; at the Lake Shore Mine, Kirkland Lake, as disseminations in quartz por-

phyry and associated with chalcopyrite and gold; at the Toburn Mine, Kirkland Lake, as a specimen of brecciated quartz and country rock with small areas of petzite and coloradoite associated with pyrite and molybdenite as a film on one surface; at the Upper Canada Mine, Gauthier Township, as small areas in contact with calaverite in sheared porphyry, associated with altaite, chalcopyrite, pyrite, and gold; at the Hollinger Mine, Porcupine district, in several specimens showing petzite as compact masses, small streaks, or disseminated throughout quartz or along cleavage planes of ankerite. In Quebec, at the Canadian Malartic Mine, Fourniere Township, as minute grains in milky quartz, associated with wehrnite, gold, and pyrite; at the Lamaque Mine, Bourlamaque Township, as an intimate intergrowth with a 4 mm. gold crystal; at the Stadacona Mine, Rouyn, as small areas in a quartz calcite gangue; at the Robb-Montbray Mine, Montbray Township, as small amounts in polished sections associated with montbrayite, melonite, frobergite, altaite, and tellurbismuth; at the Horne Mine, Noranda, Quebec, as several specimens with petzite as a coating or as compact masses associated with calaverite and coarsely crystalline altaite; at the Bevcourt Mine, Louvicourt Township, disseminated with altaite, wehrnite, and gold in massive white quartz; and at the Sullivan Consolidated Mines, disseminated with tellurbismuth in massive white quartz.

Petzite has also been reported in Canada from the Yukon Territory, British Columbia, Ontario, and Quebec. In the Yukon Territory, from the Gold Reef Claim on Gold Hill, Wheaton River district (Cairnes, 1912, p. 192); in British Columbia from the Calumet Claim, Kruger Mountain, Osoyoos Lake, Osoyoos Mining Division, and the Enterprise Claim, Long

<i>Analyses</i>	1	2	3	A
Au.....	11.10	21.56	23.691	25.42
Ag.....	49.57	34.86	41.062	41.71
Hg.....	—	6.90	—	—
Pb.....	—	0.24	0.071	—
Fe.....	0.76	0.56	trace	—
Co.....	0.76	—	—	—
As.....	1.20	—	—	—
Te.....	33.62	33.40	32.007	32.87
Insol.....	2.38	0.80	0.097	—
	<hr/> 99.39	<hr/> 98.32	<hr/> 96.928	<hr/> 100.00

1. Petzite, Hollinger Mine, Porcupine district, Ontario; incl. S, Sb, Ni, trace. Anal. Rickaby (in Walker & Parsons, 1925, p. 40). Perhaps hessite with included gold.

2. Petzite, Sylvanite Mine, Kirkland Lake, Ontario, with microscopic coloradoite; incl. Cu, S, trace, Bi, Se, none. Anal. Rickaby (in Todd, 1929, p. 81).

3. Petzite, Huronian Mine, Moss Township, Ontario (Smith, 1890, p. 439).

A. Calculated for  $\text{Ag}_3\text{AuTe}_2$ .



Lake, Greenwood Mining Division (Hoffmann, 1897, p. 12R); in Ontario from the Labine-Smith Claims, Maisonsville Township (Burrows & Hopkins, 1914, p. 35); from the McKenzie Red Lake Gold Mines, Red Lake (Hoiles, in Horwood, 1945, p. 166); the Anderson Farm, Benoit Township (Burrows, 1917, p. 250); the Sylvanite and Wright-Hargreaves Mines in the Kirkland Lake field (Todd, 1929, pp. 80-83); from the Jackson-Manion Mine, Dent Township (Bruce, 1928, p. 30); from Bigstone Bay, Lake of the Woods (Thomson, 1935, p. 48); in Quebec, from the Opasatica district (Harvie, 1912, p. 166; previously reported as sylvanite).

#### Sylvanite— $\text{AuAgTe}_4$

Monoclinic,  $P2/c$ ;  $a=8.94$ ,  $b=4.48$ ,  $c=14.59$ ,  $\beta=145^\circ 26' \pm 20'$ ;  $Z=2$  (Tunell & Ksanda, 1937).

Complex prismatic, thick tabular, skeletal or bladed crystals; imperfectly columnar to granular. Contact, lamellar, or penetration twins giving arborescent forms resembling written characters (graphic tellurium, *Schrifterz*). Silver white colour with brilliant metallic lustre, tarnishing to a light yellow. Cleavage perfect side pinakoidal; brittle;  $H=2$  (C);  $G=8.24$  (R.M.T., Kalgoorlie). Not readily distinguished from native tellurium and krennerite in fresh hand specimens.

Polished sections pleochroic (creamy-white to creamy-gray) often with marked lamellar twinning, and moderately to strongly anisotropic (light gray, brownish-gray to dark violet-gray).  $\text{HNO}_3$  (1:1) quickly stains brown, twinning becomes pronounced and a finely irregular and occasionally rectangular etch-cleavage develops when the drop is on the specimen;  $\text{HNO}_3$  (3:2) stains through iridescent to reddish-brown with development of a two directional etch-pattern in which one direction predominates;  $\text{HNO}_3$  (conc.) stains dark gray-brown, with no etch-cleavage pattern;  $\text{HCl}$ ,  $\text{KCN}$  negative;  $\text{FeCl}_3$  leaves a light yellow-brown stain (rubs clean);  $\text{KOH}$ ,  $\text{HgCl}$  negative. These observations were made on a random section. The following properties are useful in distinguishing sylvanite from calaverite, krennerite, and montbrayite in polished sections: marked pleochroism; lamellar twinning; absence of circular areas on etching with  $\text{HNO}_3$ ; the very quick reaction to various strengths of  $\text{HNO}_3$ ; the fact that an etch-cleavage pattern develops while the drop is on the specimen.

*Occurrences:* Transylvania, Western Australia, Fiji Islands, Colorado, Oregon. Sylvanite was confirmed at Nagyág, Transylvania, on a specimen of phonolite with single crystals, twins and cleavable masses of sylvanite as a thin film on one surface; at Kalgoorlie, Western Australia, as two small cleavage fragments; and at Vatukoula, Fiji Islands, as

*X-Ray Powder Pattern (R.M.T.; fig. 5)*

<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$
$\frac{1}{2}$	6.0	7.35	1	21.65	2.08	1	34.4	1.361
1	8.7	5.08	3	22.8	1.984	1	34.9	1.344
$\frac{1}{2}$	9.8	4.52	1	24.75	1.836	$\frac{1}{2}$	35.65	1.319
1	11.15	3.97	2	25.4	1.792	$\frac{1}{2}$	37.15	1.272
1	11.6	3.82	$\frac{1}{2}$	26.05	1.750	$\frac{1}{2}$	38.55	1.233
$\frac{1}{2}$	14.1	3.16	1	26.65	1.714	1	39.1	1.219
10	14.65	3.04	$\frac{1}{2}$	27.9	1.643	1	40.25	1.190
2	15.0	2.97	$\frac{1}{2}$	29.3	1.571	$\frac{1}{2}$	42.6	1.136
$\frac{1}{2}$	18.1	2.47	2	30.4	1.519	$\frac{1}{2}$	45.15	1.084
$\frac{1}{2}$	18.75	2.39	$\frac{1}{2}$	31.0	1.493	$\frac{1}{2}$	47.1	1.049
3	20.0	2.25	$\frac{1}{2}$	31.6	1.467	1	48.9	1.020
5	21.0	2.14	1	32.9	1.415	$\frac{1}{2}$	50.3	0.999
1	21.3	2.12	1	33.0	1.411			

several specimens of skeletal crystals and cleavable masses in vuggy quartz, associated with native tellurium and pyrite.

No Canadian specimen of sylvanite was available for study. Sylvanite has been reported from the Yukon Territory, Ontario, and Quebec. In the Yukon, from the Gold Reef Claim, Gold Hill, and the Buffalo Hump Group, Mount Stevens, Wheaton district (Cairnes, 1912, p. 192); in Ontario, from the Ardeen (Huronian) Mine, Moss Township, in a Royal Commission Report, but an analysis of this mineral by Smith (1890, p. 439) agrees closely with that of petzite; from Bigstone Bay, Lake of the Woods, and the Dome Mine, Porcupine district (Dana, 1944, p. 340); from the Gold Eagle Mine, Red Lake (McGill in Horwood, 1945, p. 114); from the Howey Gold Mine, Red Lake (Cornford in Horwood, 1945, p. 144); in Quebec, Harvie reported sylvanite from the Opasatica district, Pontiac County in 1910 but later revised his determination to petzite (1912, p. 166); and from the Horne Mine, Noranda (Price, 1934, p. 132).

*Tellurides of Silver***Hessite**— $\text{Ag}_2\text{Te}$ 

Crystal system unknown; the powder pattern does not index on the monoclinic elements of Tokody (1932, 1934).

Usually massive, occasionally in highly modified, distorted cubic or pseudocubic crystals, compact, or fine granular. Dark lead-gray, usually tarnishing to black, dull gray, or bronze. No cleavage but sectile with an uneven fracture.  $H = 2\frac{1}{2}$  (A);  $G = 8.41$  (R.M.T.).

Polished sections light gray, moderately anisotropic with polarization colors, gray, steel-blue, bornite-pink; characteristic confused lamellar twinning.  $\text{HNO}_3$  stains iridescent to black;  $\text{HCl}$  slowly stains black; nega-



tive on some areas; KCN on some specimens slowly stain black, on others negative;  $\text{FeCl}_3$  quickly stains iridescent; KOH negative;  $\text{HgCl}_2$  tarnishes light brown.

Hessite resembles argentite but the latter yields metallic shavings whereas hessite yields a powder when scratched.

*X-Ray Powder Pattern (R.M.T.; fig. 6)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
$\frac{1}{2}$	6.2	7.12	7	20.05	2.24	$\frac{1}{2}$	28.8	1.596
1	9.8	4.52	2	20.5	2.19	$\frac{1}{2}$	29.1	1.581
$\frac{1}{2}$	11.9	3.73	6	21.1	2.14	$\frac{1}{2}$	29.95	1.540
$\frac{1}{2}$	13.1	3.39	1	22.55	2.00	2	32.2	1.443
2	14.0	3.18	$\frac{1}{2}$	23.2	1.951	4	33.7	1.385
6	14.85	3.00	$\frac{1}{2}$	23.6	1.920	$\frac{1}{2}$	35.1	1.337
8	15.6	2.86	$\frac{1}{2}$	24.7	1.840	2	36.2	1.302
$\frac{1}{2}$	16.0	2.79	$\frac{1}{2}$	25.0	1.819	1	37.1	1.274
$\frac{1}{2}$	16.65	2.68	1	25.8	1.766	1	38.2	1.243
1	18.35	2.44	$\frac{1}{2}$	26.4	1.729	$\frac{1}{2}$	40.3	1.189
10	19.5	2.30	1	27.05	1.690			

*Occurrences:* Altai Mountains (Siberia), Transylvania, Asia Minor, Western Australia, Chile, Mexico, New Mexico, Arizona, California, Colorado, Utah. Hessite was confirmed on a specimen from Botés, Transylvania, in crystalline sphalerite with a few poorly developed crystals of hessite on a group of compact quartz crystals; and from Gold Hill, Boulder County, Colorado, as a  $5 \times 5$  mm. globular mass.

In Canada the occurrence of hessite has been established by *x*-ray powder photographs from the following localities. In the Yukon Territory from Upper Burwash Creek, as a number of small nuggets of gold and hessite from the placer workings of Mr. G. Loland; and from the Buffalo Hump Group on Mount Stevens, Wheaton River district, as sparse disseminations in quartz with galena. In British Columbia, from the Marble Bay, and Little Billie Mines, Texada Island, as minute amounts in close association with chalcopyrite, bornite, and wehrlite in polished sections; at the Harrison Group, Lindquist Lake, Omineca Mining Division, as microscopic amounts in polished sections; from the Con West property, Taseko Lake, Clinton Mining Division, as small areas embedded in chalcopyrite in polished sections; from the Pellaire Gold Mines property, Taseko Lake, Clinton Mining Division, as several small grains with free gold obtained with the superpanner; from the Lakeview and North Star Claims, Long Lake, Greenwood Mining Division, as compact masses of somewhat granular hessite  $20 \times 20$  mm. with a few small quartz crystals; from the Boundary District, as a few small isolated fragments; and from

the Hedley Monarch Mine, Osoyoos Mining Division, as a  $\frac{1}{2}$  mm. area in a polished section, in contact with altaite and petzite. In Ontario, from the Ardeen (Moss) Mine, Moss Township, as grains of hessite and altaite in a gangue of quartz and calcite; and also, together with petzite, as a massive black crust, 5 mm. thick, on one surface of vein quartz with gold and hessite in the form of  $\frac{1}{2}$  mm. bronzy to black films partially covering two other surfaces; at the Hollinger Mine, Porcupine district, as thin films on cleavage planes of ankerite; at a prospect in Tisdale Township, Porcupine area, as a specimen of vein quartz with a 4 mm. area of hessite and gold at the contact of a serpentine stringer; and at the Kirkland Lake Mine, Kirkland Lake, as coarsely crystalline calcite with grains of hessite associated with small compact masses of coloradoite, chalcopyrite and tetrahedrite. In Quebec at the Sullivan Mine, Bourlamaque Township, as disseminated grains in quartz and associated with tourmaline, pyrite, chalcopyrite and gold.

Hessite has also been reported elsewhere in Canada, from the Yukon Territory, British Columbia, Ontario, and Quebec. In the Yukon Territory from the Gold Reef Claim on Gold Hill, Wheaton River district (Cairnes, 1912, p. 192); in British Columbia from the Calumet Claim, Kruger Mountain, Osoyoos Mining Division (Hoffmann, 1897, p. 12R); from Liddle Creek, Kaslo River, W. Kootenay district (Hoffmann, 1897, p. 10R); in Ontario, from Gold Creek, Pine Portage Bay, Lake of the Woods (Coleman, 1896, p. 105); from the Powell Claim, Deloro Township (Burrows, 1912, p. 229); from Bigstone Bay, Lake of the Woods (Thomson, 1935, p. 48); from the Toburn (Tough-Oakes) Mine, Kirkland Lake (Campbell & Deyell, in Burrows & Hopkins, 1914, p. 23); in Quebec from the McWatters Mine, Rouyn Township (Hawley, in Thomson, 1935, p. 48); and the Horne Mine, Noranda (Price, 1934, p. 132).

<i>Analyses</i>	1	2	3	4	A
Au.....	2.29	—	0.10	trace	—
Ag.....	60.68	61.01	61.88	62.01	62.86
Te.....	37.33	35.40	—	29.62	37.14
Fe.....	—	—	—	1.76	—
	100.30	96.41		93.39	100.00

1. Hessite, Lakeview Claim, Long Lake, Greenwood Mining Division, B. C. Anal. Guess & Guess (in Hoffman, 1897, p. 12R).

2. Hessite, Gold Creek, Pine Portage Bay, Lake of the Woods, Ontario. Anal. Lawson (in Coleman, 1896, p. 105).

3. Hessite, Powell Claim, Deloro Township, Ontario (Burrows, 1912, p. 229).

4. Hessite, Huronian Mine, Moss Township, Ontario (Smith, 1890, p. 440).

A. Calculated for  $\text{Ag}_2\text{Te}$ .



**Empressite**— $\text{Ag}_5\text{Te}_3$ <sup>4</sup>

Massive to finely granular, with no indication of crystal form. Heavy metallic lustre and an almost jet black colour, like petzite but with a slight bronzy cast. No cleavage, but with a finely conchoidal to uneven fracture;  $H=3\frac{1}{2}$  (C);  $G=7.61\pm0.01$  (R.M.T.).

Polished sections give a smooth surface which shows strong reflection pleochroism (light gray-mauve to creamy-white, depending on the orientation) and intense anisotropism (white, yellowish green, russet-brown, brown, dark blue). The mineral makes a solid moderately coarse-grained mosaic.  $\text{HNO}_3$ , slow effervescence and slow iridescent stain which may be removed by hard buffing;  $\text{HCl}$ ,  $\text{KCN}$ , negative;  $\text{FeCl}_3$ , iridescent stain which remains on buffing;  $\text{KOH}$ , negative;  $\text{HgCl}_2$ , quick iridescent stain which remains on buffing.

*X-Ray Powder Pattern* (R.M.T.; fig. 7)

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
$\frac{1}{2}$	10.1	4.38	1	20.1	2.24	$\frac{1}{2}$	29.9	1.542
$\frac{1}{2}$	11.2	3.96	10	20.8	2.16	$\frac{1}{2}$	31.8	1.459
1	12.5	3.55	2	21.3	2.12	$\frac{1}{2}$	32.25	1.441
$\frac{1}{2}$	13.1	3.39	1	22.2	2.03	$\frac{1}{2}$	32.65	1.425
$\frac{1}{2}$	13.8	3.22	1	23.5	1.928	2	34.8	1.347
2	14.7	3.03	$\frac{1}{2}$	23.8	1.905	$\frac{1}{2}$	35.8	1.314
$\frac{1}{2}$	15.8	2.82	$\frac{1}{2}$	24.3	1.868	$\frac{1}{2}$	36.6	1.289
1	17.0	2.63	$\frac{1}{2}$	27.6	1.659	$\frac{1}{2}$	37.0	1.277
5	17.6	2.54						

*Occurrences:* This mineral has been found only in Colorado. A specimen from the Empress Josephine Mine, Kerber Creek District (type locality), is a compact mass of empressite partially covered with a coating of a clay-like material with disseminated yellowish brown sphalerite and finely crystalline pyrite; on one corner is a small area of galena. Established also at the Red Cloud Mine, Boulder County (mislabelled petzite), as sparse disseminations in rock with altaite.

*Tellurides of Copper***Weissite**— $\text{Cu}_2\text{Te}$ 

Pseudocubic;  $a=7.22$  (R.M.T.);  $Z=8$ .

Massive with irregular fracture. Bluish-black tarnishing to black. No cleavage;  $H=3$  (B);  $G\approx6$ .

Polished sections light gray with moderate anisotropism (pink, bluish-

<sup>4</sup> This composition is indicated by pyrosyntheses.

gray to blue), like chalcocite. The mineral is sponge-like with gangue filling the cavities.  $\text{HNO}_3$  effervesces and stains brown;  $\text{HCl}$  stains light-brown;  $\text{KCN}$  stains light-brown;  $\text{FeCl}_3$  stains brown;  $\text{KOH}$  slowly stains light-brown;  $\text{HgCl}_2$  stains light-brown.

*X-Ray Powder Pattern (R.M.T.; fig. 8)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
2	6.1	7.23	5	22.7	1.992	1	34.3	1.364
10	12.15	3.65	2	25.15	1.809	2	35.0	1.340
7	13.85	3.21	1	26.0	1.754	$\frac{1}{2}$	37.0	1.277
1	16.4	2.72	1	28.4	1.616	1	39.8	1.201
2	17.45	2.56	$\frac{1}{2}$	29.15	1.578	2	40.3	1.189
2	19.7	2.28	$\frac{1}{2}$	29.9	1.542	1	42.3	1.142
2	20.7	2.17	3	32.1	1.447	$\frac{1}{2}$	43.0	1.127
4	21.6	2.09	$\frac{1}{2}$	32.9	1.415	$\frac{1}{2}$	45.0	1.087

*Occurrences:* Colorado, Japan. The only material available for study was a small chip of tarnished weissite disseminated through gangue, from the Good Hope Mine, Vulcan, Colorado. This mineral has not been found in Canada.

**Rickardite**— $\text{Cu}_3\text{Te}_2$

Tetragonal,  $P4/nmm$ ;  $a=3.97$ ,  $c=6.11$  (Peacock);  $Z=1$ .

Not observed with crystal form; massive with an irregular fracture. Deep purplish-red tarnishing to a purplish-brown. Brittle with  $H=3\frac{1}{2}$  (B);  $G=7.54$ .

Polished sections give a finely pitted surface; purplish-red with strong reflection pleochroism (blue-gray to purplish-red), and intense anisotropism (white, blue-gray, dark blue, fiery orange).  $\text{HNO}_3$  blackens with effervescence;  $\text{HCl}$  etches gray to brown;  $\text{KCN}$  slowly bleaches gray;  $\text{FeCl}_3$  bleaches gray to brown;  $\text{KOH}$  tarnishes iridescent to black;  $\text{HgCl}_2$

*X-Ray Powder Pattern (R.M.T.; fig. 9)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
$\frac{1}{2}$	7.3	6.05	4	22.8	1.984	$\frac{1}{2}$	36.5	1.292
6	13.25	3.35	1	25.05	1.816	$\frac{1}{2}$	37.8	1.254
1	14.70	3.03	2	26.85	1.703	$\frac{1}{2}$	38.7	1.229
2	15.90	2.81	$\frac{1}{2}$	27.5	1.665	$\frac{1}{2}$	39.5	1.209
4	17.65	2.54	1	30.2	1.528	3	41.7	1.156
$\frac{1}{2}$	18.5	2.42	2	32.75	1.421	1	43.3	1.121
10	21.8	2.07	2	33.2	1.404	1	45.2	1.083
1	22.0	2.05	1	35.05	1.339	$\frac{1}{2}$	48.0	1.034



slowly tarnishes purple. The mineral has a fine-granular appearance with crossed nicols but large irregular lath-like areas extinguish simultaneously. Readily distinguished by its colour and anisotropism.

*Occurrences:* Colorado, Arizona, Salvador, Australia, Japan. The foregoing properties were obtained on a compact lens of tarnished rickardite, 30×10 mm., in quartz from the Good Hope Mine, Vulcan, Colorado.

In Canada, Price (1934) lists rickardite? amongst the telluride minerals occurring at the Horne Mine, Noranda, Quebec, but without details.

### *Tellurides of Nickel and Iron*

#### **Melonite**—NiTe<sub>2</sub>

Hexagonal,  $C\bar{3}m$ ;  $a=3.835$ ,  $c=5.255$ ;  $Z=1$  (Peacock & Thompson, 1946a).

Rounded tabular hexagonal crystals and foliated masses. Light steel-gray to tin white with a reddish cast, tarnishing through yellow to bronze-red and light-brown. Cleavage eminent basal, giving highly flexible flakes.  $H=2$  (B);  $G=7.72$ .

Polished sections light pink, particularly striking against the pure white of tellurbismuth; distinctly coppery coloured when observed in reflected light under a binocular. Stands out in relief against altaite, tellurbismuth, and montbrayite. Moderately anisotropic (grayish mauve to yellowish brown).  $HNO_3$  instantly effervesces and stains black;  $HCl$ ,  $KCN$ , negative;  $FeCl_3$  slowly stains light-brown and brings out scratches (rubs off);  $KOH$ ,  $HgCl_2$ , negative.

*X-Ray Powder Pattern* (Peacock & Thompson, 1946a; fig. 10)

<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)	<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)	<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)
1	8.4	5.26	2	29.0	1.586	$\frac{1}{2}$	42.6	1.136
1	13.4	3.32	6	29.85	1.544	$\frac{1}{2}$	43.9	1.109
10	15.85	2.81	1	33.2	1.404	2	45.3	1.081
3	17.0	2.63	1	35.9	1.311	1	48.8	1.022
5	22.0	2.05	2	38.9	1.224	1	50.2	1.001
5	23.7	1.912	1	39.8	1.201			

*Occurrences:* Colorado, California, South Australia.

In specimens from the Cresson Mine, Cripple Creek, Colorado, melonite was observed as flat thin bronzy tarnished plates in intimate intergrowth with calaverite and gold.

In Canada, the occurrence of this rare telluride has been established at the Robb-Montbray Mine, Montbray Township, Quebec, and described in detail (Peacock & Thompson, 1946a). Here the nickel telluride occurs as small patches (up to 20 mm. wide) associated with rich compact masses of altaite, tellurbismuth, montbrayite, pyrite, chalcopyrite, and gold, or

disseminated through rock. In polished sections it occurs as small rounded and distorted hexagonal sections sometimes enclosing and sometimes enclosed in an intergrowth of altaite and tellurbismuth. Also as thin selvages, together with frobergite, surrounding chalcopyrite, and intimately associated with chalcopyrite, petzite, gold, montbrayite, and frobergite. At the Wright-Hargreaves Mine, Kirkland Lake, Ontario, as a small area ( $3 \times 3$  mm.) in contact with gold in prophyry; at the Toburn (Tough-Oakes) Mine, Kirkland Lake, associated with altaite and petzite in polished section; and at the Macassa Mine, Kirkland Lake, as small ( $2 \times 2$  mm.) areas in contact with gold in brecciated vein material.

#### Frobergite— $\text{FeTe}_2$

Orthorhombic,  $Pmn$ ;  $a=3.85$ ,  $b=5.28$ ,  $c=6.26$ ;  $Z=2$  (Thompson, 1947).

Not observed in hand specimens. Polished sections pink; purplish pink against melonite which appears yellowish-pink by contrast. Strongly anisotropic (orange-red to inky blue).  $H=C+G=7.98$  (calc).  $\text{HNO}_3$ , rapid effervescence and a black etch;  $\text{HCl}$ ,  $\text{KCN}$ ,  $\text{FeCl}_3$ ,  $\text{KOH}$ ,  $\text{HgCl}_2$ , negative.

#### X-Ray Powder Pattern (R.M.T.; fig. 11)

<i>I</i>	$\theta(\text{Fe})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Fe})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Fe})$	$d(\text{meas.})$
3	17.1	3.29	4	31.65	1.841	1	49.4	1.273
2	18.0	3.13	$\frac{1}{2}$	32.65	1.791	1	52.3	1.221
10	20.2	2.80	1	33.75	1.739	2	53.55	1.201
7	20.95	2.70	$\frac{1}{2}$	34.65	1.699	2	56.2	1.163
1	21.45	2.64	3	37.9	1.573	1	58.25	1.136
$\frac{1}{2}$	23.3	2.44	1	38.45	1.554	$\frac{1}{2}$	59.6	1.120
$\frac{1}{2}$	25.75	2.22	$\frac{1}{2}$	39.1	1.532	3	61.0	1.105
5	27.95	2.06	2	39.75	1.511	$\frac{1}{2}$	62.85	1.086
$\frac{1}{2}$	28.65	2.01	2	45.85	1.347	2	64.4	1.071
3	29.95	1.935	$\frac{1}{2}$	47.1	1.319	1	70.7	1.024

*Occurrence:* This new mineral was recognized and described as small areas in association with altaite, tellurbismuth, montbrayite, melonite, petzite, chalcopyrite, pyrite, marcasite, sphalerite, chalcocite, covellite, and free gold in several polished sections of ore from the Robb-Montbray Mine, Montbray Township, Quebec (Thompson, 1947). It occurs most commonly as complete or embayed zones up to 50 microns wide surrounding chalcopyrite and making contact with altaite, gold, or melonite; less commonly as small irregular areas in gold, petzite, or chalcopyrite; once noted as an intimate intergrowth with gold, petzite, and montbrayite.

*Tellurides of Lead and Mercury***Altaite**—PbTe

Cubic,  $Fm\bar{3}m$ ;  $a = 6.430$  (R.M.T.);  $Z = 4$ .

Rarely in cubes and octahedrons but usually finely granular to massive. Tin white with a grayish-green cast and high metallic lustre, tarnishing bronze-yellow, bright blue-green, dull blue-gray to black; once observed tarnished bronze-red. Cleavage cubic, usually poor but sometimes perfect. Somewhat sectile;  $H = 3$  (C);  $G = 8.19$  (R.M.T.).

Polished sections pure white, but bluish against tellurbismuth which has a pink cast, and white against galena which appears mauvish-gray by contrast; isotropic.  $HNO_3$  effervesces and stains iridescent to dark gray; HCl tarnishes iridescent; KCN negative;  $FeCl_3$  quickly tarnishes iridescent; KOH,  $HgCl_2$  negative.

*X-Ray Powder Pattern* (R.M.T.; fig. 12)

<i>I</i>	$\theta$ (Cu)	<i>d</i> (meas.)	<i>I</i>	$\theta$ (Cu)	<i>d</i> (meas.)	<i>I</i>	$\theta$ (Cu)	<i>d</i> (meas.)
1	11.9	3.73	2	28.6	1.606	2	49.1	1.017
10	13.8	3.22	5	32.3	1.439	2	52.6	0.968
8	19.7	2.28	4	35.9	1.311	1	59.3	0.894
1	23.5	1.928	1	42.6	1.136	2	63.4	0.860
3	24.5	1.854	2	45.9	1.070			

*Occurrences:* Altai Mountains (Siberia), Transylvania, Burma, Western Australia, Chile, California, New Mexico, Colorado, North Carolina. At Nagyág, Transylvania, altaite was observed as irregular inclusions in coarse nagyagite with rhodochrosite; at Las Cruces, New Mexico, as disseminated nests in coarsely crystalline barite; at the Empress Josephine Mine, Kerber Creek district, Colorado, as small inclusions in empressite; at the Red Cloud Mine, Boulder County, Colorado, as disseminated grains associated with pyrite and tarnished empressite in porphyry.

In Canada, the occurrence of altaite has been confirmed by  $x$ -ray powder photographs in Yukon Territory, at Upper Burwash Creek, associated with hessite, gold, and hedleyite, in polished section; in British Columbia, at the Charley Group and Hido Group (Pellaire Mines Ltd.), near Taseko Lake narrows, Clinton Mining Divisions, in vuggy limonitic quartz associated with hessite, gold, and several sulphides; near Glacier Lake, New Westminster Mining Division, as small nests in float of limonitic vein-calcite; at the Hedley Monarch Mine, Osoyoos Mining Division, associated with hessite and petzite, in polished section; in Manitoba, at Copper Lake, as grains in quartz; in Ontario, at the Ardeen (Moss) Mine, Thunder Bay district, with hessite in a quartz-calcite



gangue; at the Kirkland Golden Gate Mine, Swastika, as a finely crystalline mass associated with calaverite and free gold coating one surface of white pyritic quartz; at the Lake Shore, Macassa, Toburn (Tough-Oakes), Kirkland Lake, Wright-Hargreaves, Teck-Hughes, Sylvanite, Bidgood, and Upper Canada Mines, Kirkland Lake Area, as disseminated grains or compact masses up to  $40 \times 15$  mm., embedded in dark silicified rock, quartz, or altered syenite porphyry, and associated with coloradoite, petzite, calaverite, melonite, pyrite, molybdenite, chalcopyrite, and gold; at the Dome Mine, South Porcupine, as scattered grains associated with pyrrhotite in quartz; in Quebec, at the Robb-Montbray Mine, Montbray Township, in solid rich masses of tellurides, sulphides, and gold, with altaite in substantial masses, usually intergrown with tellurbismuth and altering to an earthy blue-gray coating; at the Horne Mine, Noranda, with petzite and calaverite in coarsely crystalline masses, or intergrown with tellurbismuth; and at the Bevcourt Mine, Louvicourt Township, disseminated with petzite wehrnite, and gold in massive white quartz.

In addition, altaite has been reported in Canada by Hoffmann (1895, p. 29; 1897, p. 10), Cairnes (1912, p. 197), Todd (1929, p. 74), and Thomson (1937); in British Columbia at the Lakeview Claims, Long Lake Camp, Greenwood Mining Division; from the Rhoderic Dhu Claim in the same camp; from Liddle Creek, Kaslo River, W. Kootenay district; and from the Pay Roll Mine, near Cranbrook, Fort Steele Mining Division; in Ontario, from the Three Ladies Mine, Lake of The Woods district; and the Ashley Mine, Bannockburn Township; at the Gold Eagle Mine, Red Lake (McGill, in Horwood, 1945, p. 114); at the Howey Gold Mine, Red Lake (Cornford, in Horwood, 1945, p. 144); at the McKenzie Red Lake Gold Mines, Red Lake (Hoiles, in Horwood, 1945, p. 166); and at the Chambers-Ferland Group, Schreiber (Thomson, 1923, p. 39).

<i>Analyses</i>	1	2	3	A
Pb.....	49.72	61.26	57.33	61.91
Au.....	0.01	—	} 1.10	—
Ag.....	2.09	—		—
Cu.....	—	0.20	1.60	—
Fe.....	0.63	0.64	1.63	—
Te.....	39.57	36.84	35.66	38.09
S.....	—	0.29	1.95	—
Insol.....	7.84	0.46	0.38	—
	99.86	99.69	99.65	100.00

1. Altaite, Lakeview Claim (mean of two analyses), associated with hessite, gold, native copper, native tellurium. Anal. Johnston (in Hoffmann, 1897, p. 11).

2. Altaite, Lake Shore Mine, spherical masses associated with coloradoite; incl. Hg trace, Bi, Se, none. Anal. Rickaby (in Todd, 1929, p. 74).

3. Altaite, Lake Shore Mine, associated with chalcopyrite in nests and veinlets; incl. Hg, Bi, Se, none. Anal. Rickaby (in Todd, 1929, p. 75).

A. Calculated for PbTe.

**Nagyagite**— $\text{Au(Pb, Sb, Fe)}_8(\text{Te, S})_{11}$ 

Tetragonal,  $P\bar{4}$ ;  $a=4.14$ ,  $c=30.15$  X;  $Z=1$  (Berry, 1946).

Squarish basal plates and folia with rectangular striations; also granular massive. Blackish lead-gray with high metallic lustre. Cleavage basal, giving bent flexible flakes;  $H=1\frac{1}{2}$  (B—);  $G=7.49$ .

Polished sections difficult to make; gray-white distinctly anisotropic (lead-gray to dark blue) showing twinning; often intergrown with altaite.  $\text{HNO}_3$  slowly stains iridescent;  $\text{HCl}$ ,  $\text{KCN}$ ,  $\text{FeCl}_3$ ,  $\text{KOH}$ ,  $\text{HgCl}_2$  negative.

*X-Ray Powder Pattern* (R.M.T.; fig. 13)

<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$
$\frac{1}{2}$	12.2	3.64	3	18.5	2.42	3	24.9	1.826
$\frac{1}{2}$	13.05	3.40	$\frac{1}{2}$	19.5	2.30	2	28.85	1.702
10	14.75	3.02	$\frac{1}{2}$	20.55	2.19	$\frac{1}{2}$	27.7	1.654
$\frac{1}{2}$	15.2	2.93	4	21.7	2.08	3	30.7	1.506
6	15.8	2.82	$\frac{1}{2}$	22.0	2.05	1	31.7	1.463
1	16.9	2.64	1	23.8	1.905			

*Occurrences*: Transylvania, Japan, Western Australia, New Zealand, Fiji Islands, California, Colorado, North Carolina, Montana.

In Canada, nagyagite has been reported from British Columbia and Ontario, but these occurrences require confirmation. Kemp (1898, p. 317) reported nagyagite from the Olive Mabel Claim, Gainor Creek, Lardeau Mining Division, B. C. Coleman (1896, p. 106) speaks of "a telluride of lead, probably nagyagite, having been obtained at the Huronian Mine [Moss Township, Thunder Bay district, Ontario] as reported by Dr. Ellis of the School of Science, Toronto."

**Coloradoite**— $\text{HgTe}$ 

Cubic,  $F\bar{4}3m$ ;  $a=6.440$  (R.M.T.);  $Z=4$ .

Crystals unknown, usually massive and sometimes granular. Bright iron-black tarnishing to a diagnostic dull purple, which distinguishes it in hand specimens from petzite and tetrahedrite. No cleavage, but brittle with a sub-conchoidal fracture.  $H=2\frac{1}{2}$  (C);  $G=8.10$  (R.M.T.).

Polished sections pinkish-gray, resembling tetradedrite; isotropic.  $\text{HNO}_3$  slowly stains brown, some areas almost negative;  $\text{HCl}$ ,  $\text{KCN}$ , negative;  $\text{FeCl}_3$  tarnishes iridescent; after cleaning with  $\text{HCl}$ , the surface shows moderate anisotropism (dark brownish-red to blue-gray);  $\text{KOH}$ ,  $\text{HgCl}_2$ , negative. Distinguished from other tellurides in polished section by its pinkish-gray colour and resistance to etching with  $\text{HNO}_3$ .

*Occurrences*: Colorado, California, Western Australia. At the Smuggler Mine, Boulder County, Colorado, tarnished coloradoite was observed in

*X-Ray Powder Pattern (R.M.T.; fig. 14)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
10	11.9	3.73	4	35.8	1.314	$\frac{1}{2}$	55.9	0.928
1	13.8	3.22	3	38.3	1.240	1	58.5	0.903
9	19.7	2.28	1	42.5	1.138	2	63.3	9.860
7	23.3	1.943	3	45.0	1.087	1	66.5	0.839
2	28.5	1.611	1	49.1	1.107			
3	31.3	1.479	$\frac{1}{2}$	51.6	0.982			

a chip of gangue; at Kalgoorlie, Western Australia, as small chips with attached petzite, and krennerite, both free and attached to gangue.

In Canada, coloradoite has been found only in Ontario and Quebec, and has been confirmed at the Lake Shore Mine, Kirkland Lake, Ontario, as compact masses up to 20×30 mm. associated with altaite; at the Wright-Hargreaves Mine, Kirkland Lake, as compact masses with gold, embedded in quartz; at the Toburn (Tough-Oakes) Mine, Kirkland Lake, as lenses up to 10×5 mm. associated with altaite, pyrite and chalcopyrite in quartz stringers in syenite porphyry; at the Bidgood Mine, Kirkland Lake, as compact masses of exceptional size associated with calaverite, altaite, gold, and molybdenite in brecciated quartz; at the Kirkland Lake Mine, as small compact masses embedded in coarsely crystalline calcite and associated with hessite, chalcopyrite, and tetrahedrite; and at the Hollinger Mine, Timmins, as several fragments of coarsely crystalline calcite with films of hessite, coloradoite, tetrahedrite, and chalcopyrite.

In addition coloradoite has been reported from the Teck-Hughes and Sylvanite Mines, Kirkland Lake (Todd, 1929, pp. 82–83); from the Ardeen (Moss) Mine, Moss Township (Thomson, 1931, p. 52); and the Robb-Montbray Mine, Montbray Township, Quebec (Thomson, 1928, p. 13).

<i>Analyses</i>	1	A
Hg.....	58.55	61.14
Pb.....	1.60	—
Te.....	39.10	38.86
Insol.....	0.25	—
	99.50	100.00

1. Coloradoite, Lake Shore Mine, with a small amount of altaite; incl. Au, Ag, Bi, Se, Cu, Fe, S, none. Anal. Rickaby, in Todd (1929, p. 80).

2. Calculated for HgTe.

*Tellurides of Bismuth***Hedleyite**—Bi<sub>7</sub>Te<sub>8</sub>

Hexagonal,  $R\bar{3}m$ ;  $a=4.46$ ,  $c=118.8$ ;  $r=39.68$ ,  $\alpha=6^\circ 26'$ ;  $Z=2$  (Warren & Peacock, 1945).



In plates giving flexible and slightly elastic folia; occasionally a hint of crystal form. Tin-white with an iron-black tarnish; perfect basal cleavage  $H=2$  (A);  $G=8.91$ .

Polished sections white, slightly anisotropic (light to dark gray) on nearly basal section.  $HNO_3$  effervesces and quickly turns gray; fumes give brown iridescent tarnish;  $HCl$ ,  $KCN$ , negative;  $FeCl_3$  stains brown to iridescent;  $KOH$ ,  $HgCl_2$  negative.

*X-Ray Powder Pattern* (Peacock; fig. 15)

<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)	<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)	<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)
1	10.1	4.38	1	21.4	2.11	3	32.8	1.419
$\frac{1}{2}$	12.3 $\beta$	3.26	3	22.8	1.984	2	33.6	1.389
10	13.7	3.25	3	24.7	1.840	2	36.0	1.308
5	19.0	2.36	4	28.3	1.621	1	36.65	1.288
4	20.15	2.23	4	31.3	1.480			

*Occurrences:* This species was described by Warren & Peacock (1945) from the Good Hope mineral claim, Hedley, British Columbia. Hedleyite occurs as thick plates often intercalated with joseite B, native bismuth, and gold, and associated with arsenopyrite, molybdenite, and pyrrhotite, in quartz and skarn. A second occurrence of this rare mineral was established at Upper Burwash Creek, Kluane Lake district, Yukon Territory. A polished section of a nugget of gold and hessite showed small amounts of altaite and several laths of a bismuth telluride in the hessite; these laths gave the hedleyite pattern.

<i>Analyses</i>	1	2	A
Bi. ....	80.60	81.55	79.3
Te. ....	18.52	17.60	20.7
S. ....	0.12	0.04	—
	99.24	99.19	100.0

1, 2. Hedleyite, Hedley, British Columbia. Anal. Eldridge (in Warren & Peacock, 1945). In anal. 1 also spectrographic Sb 0.05, Pb 0.01, Cu 0.01.

A. Calculated for  $Bi_7Te_3$ .

#### Joseite A— $Bi_{4+x}Te_{1-x}S_2$

Hexagonal,  $R\bar{3}m$ ;  $a=4.24$ ,  $c=39.69$ ;  $r=13.45$ ,  $\alpha=18^\circ 08'$ ;  $Z=1$  (Peacock, 1941).<sup>5</sup>

Sheets and plates with occasional straight edges yielding soft, flexible,

<sup>5</sup> Following Peacock (1941, p. 102) the general formula  $Bi_{4+x}(Te, S)_{3-x}$ , omitting Se as unessential, is accepted for joseite. The two types of joseite noted by Peacock,  $Bi_{4+x}Te_{1-x}S_2$  and  $Bi_{4+x}Te_{2-x}S$ , have been recognized in further specimens and they are here denoted as joseite A and joseite B, respectively.

inelastic folia; meagre evidence of crystal form. Silver-white colour with high metallic lustre tarnishing lead gray, iridescent, steel-blue to iron-black. Perfect basal cleavage;  $H=2$  (A-B);  $G=8.10$ .

Polished sections less white than galena with hardness B on basal sections, and A on transverse sections. Basal sections nearly isotropic; transverse sections moderately anisotropic in greenish-gray to dark greenish-gray. Etch-reactions:  $HNO_3$  effervesces and etches dark gray; HCl stains light gray; KCN negative;  $FeCl_3$  stains pale blue-gray; KOH negative;  $HgCl_2$  negative.

*X-Ray Powder Pattern* (Peacock, 1941; fig. 16)

<i>I</i>	$\theta$ (Cu)	<i>d</i> (meas.)	<i>I</i>	$\theta$ (Cu)	<i>d</i> (meas.)	<i>I</i>	$\theta$ (Cu)	<i>d</i> (meas.)
2	10.1	4.38	$\frac{1}{2}$	25.0	1.819	2	39.45	1.210
2	12.3	3.61	$\frac{1}{2}$	25.6	1.779	$\frac{1}{2}$	40.8	1.176
$\frac{1}{2}$	13.0 $\beta$	3.09	3	26.15	1.744	$\frac{1}{2}$	47.4	1.044
1	13.45	3.30	1	27.7	1.654	1	48.5	1.026
10	14.5	3.07	$\frac{1}{2}$	28.4	1.616	2	49.9	1.005
2	17.4	2.57	3	30.0	1.537	$\frac{1}{2}$	51.5	0.982
5	20.1	2.24	2	33.05	1.409	1	53.5	0.956
5	21.4	2.11	3	34.85	1.345	1	54.6	0.943
1	22.0	2.05	1	36.2	1.302	$\frac{1}{2}$	57.9	0.907
1	23.0	1.967	2	38.1	1.246	2	66.9	0.836
2	23.95	1.894	$\frac{1}{2}$	39.0	1.221	2	70.4	0.816

*Occurrences:* Brazil, Spain, England. In Canada, joseite A has been positively identified in British Columbia at Glacier Gulch, Hudson Bay Mountain, near Smithers, where it occurs as coarse plates up to  $\frac{1}{2}$  inch wide and 2 inches long, sometimes intergrown with bismuth and joseite B; and at the Windpass Mine, near Chu Chua, where it is intimately associated with bismuthinite.

<i>Analyses</i>	1	2	A
Bi.....	79.3	82.7	81.4
Te.....	12.2	12.0	12.4
S.....	6.0	6.0	6.2
Se.....	none	none	—
Au.....	trace	—	—
Insol.....	trace	—	—
	97.5	100.7	100.0

1. Joseite A, Glacier Gulch, Hudson Bay Mountain, near Smithers, Omineca Mining Division, B. C. Anal. Forward (in Warren & Davis, 1940, p. 110).

2. Joseite A ("auriferous tetradymite," ROM, M 17255), Glacier Gulch, Hudson Bay Mountain, near Smithers, Omineca Mining Division, B. C. Anal. Meen (in Peacock, 1941, p. 92).

A. Calculated for  $Bi_4TeS_3$ .

**Joseite B**— $\text{Bi}_{4+x}\text{Te}_{2-x}\text{S}$ 

Hexagonal,  $R\bar{3}m$ ;  $a=4.33$ ,  $c=40.75$ ;  $r=13.81$ ,  $\alpha=18^\circ 02'$ ;  $Z=1$  (Peacock, 1941).

Identical with joseite A in most physical properties, and distinguished only by accurate specific gravity measurements.  $G=8.3$ .

*X-Ray Powder Pattern* (R.M.T.; fig. 17)

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
2	9.8	4.52	4	19.6	2.29	2	29.4	1.566
1	12.05	3.69	5	20.8	2.16	1	32.2	1.443
$\frac{1}{2}$	12.7 $\beta$	3.16	1	21.3	2.12	2	34.0	1.375
$\frac{1}{2}$	13.05	3.40	$\frac{1}{2}$	22.2	2.03	1	37.1	1.274
10	14.1	3.16	2	23.3	1.943	1	38.1	1.246
1	16.95	2.64	3	25.6	1.779			

*Occurrences:* Brazil. In Canada the occurrence of joseite B has been confirmed in British Columbia at Glacier Gulch, Hudson Bay Mountain, near Smithers, where it is associated with bismuth and joseite A; and at the Good Hope mineral claim, near Hedley, Osoyoos Mining Division, where it occurs as coarse plates often intergrown with native bismuth and associated with hedleyite, pyrrhotite, arsenopyrite, molybdenite, and gold, in quartz and skarn.

<i>Analysis</i>	1	A
Bi.....	75.14	64.4
Te.....	19.25	22.7
S.....	3.64	2.9
Pb.....	0.68	—
Fe.....	0.52	—
Insol.....	0.30	—
	99.53	100.0

1. Joseite B, Glacier Gulch, Hudson Bay Mountain, near Smithers, Omineca Mining Division, B. C. Anal. Forward & Lyle.

A. Calculated for  $\text{Bi}_4\text{Te}_2\text{S}$ .

**Tellurbismuth**— $\text{Bi}_2\text{Te}_3$ 

Hexagonal,  $R\bar{3}m$ ;  $a=4.375$ ,  $c=30.39$ ;  $r=10.44$ ,  $\alpha=24^\circ 11\frac{1}{2}'$ ;  $Z=1$  (Peacock, in Peacock & Berry, 1940, p. 67).

Plates and foliated masses with splendid metallic lustre; colour lead gray with a pinkish cast, tarnishing dull gray, black, or iridescent. Perfect basal cleavage yielding flakes slightly less flexible than those of tetradyte; inelastic, and somewhat sectile;  $H=2$  (B);  $G=7.81 \pm 0.10$ .

Polished sections pinkish-white in reflected light, with hardness A transverse to the cleavage and slightly harder on cleavage surfaces.



Weakly anisotropic (yellow to dark gray). The author was unable to duplicate his etch-reactions on x-rayed specimens of tellurbismuth; however, the mineral, like all the bismuth tellurides, is always positive to  $\text{HNO}_3$  and  $\text{FeCl}_3$ . It is felt that these minerals cannot be distinguished with certainty on the basis of etch-reactions.

Tellurbismuth is frequently intergrown with tetradymite or altaite. Frondel (1940) showed that "vandiestite" was a mixture of tellurbismuth and hessite. Harcourt (1942, p. 106) gives a list of powder spacings and intensities for "vandiestite" from Colorado. These spacings and intensities agree very closely with patterns of an intergrowth of tellurbismuth and altaite from the Robb-Montbray Mine, Montbray Township, Quebec.

*X-Ray Powder Pattern (R.M.T.; fig. 18)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
1	8.75	5.05	$\frac{1}{2}$	29.45	1.568	1	43.45	1.118
$\frac{1}{2}$	11.75	3.77	4	31.15	1.486	$\frac{1}{2}$	44.75	1.092
10	13.85	3.21	$\frac{1}{2}$	31.95	1.450	1	45.85	1.071
$\frac{1}{2}$	16.55	2.69	2	33.05	1.410	2	47.35	1.045
8	18.95	2.37	2	33.45	1.394	$\frac{1}{2}$	47.75	1.038
1	20.15	2.23	1	35.05	1.339	$\frac{1}{2}$	48.55	1.025
4	20.55	2.19	2	36.35	1.297	$\frac{1}{2}$	49.15	1.016
4	22.25	2.03	1	37.35	1.267	1	50.6	0.995
$\frac{1}{2}$	22.65	1.996	1	38.05	1.247	$\frac{1}{2}$	52.85	0.964
3	25.15	1.809	1	39.95	1.207	$\frac{1}{2}$	53.95	0.951
1	26.95	1.696	1	40.4	1.186	$\frac{1}{2}$	55.85	0.928
3	28.55	1.608	2	41.65	1.157	1	56.65	0.920

*Occurrences:* Japan, New Mexico, Georgia, Montana, California, and Sweden. At Boliden, Sweden, as a coarsely crystalline compact mass (40×30 mm.) of tellurbismuth and tetradymite.

In Canada, tellurbismuth has been positively identified in the Yukon Territory, British Columbia, Ontario, and Quebec. In the Yukon Territory, as several flat plates from the placer workings of Mr. G. Loland, Upper Burwash Creek, Kluane Lake district; in British Columbia, from the Hunter Group, Khutze Inlet, Skeena Mining Division, as several plates of tellurbismuth; from the Ashloo Mine, near Squamish, Vancouver Mining Division, as several plates of tellurbismuth; and from the Hedley Yuniman Gold Fields Ltd., Bradshaw Creek, Osoyoos Mining Division, as small plates of tellurbismuth and polished sections; in Ontario, from the Ardeen Mine, Moss Township, in a polished section with chalcopyrite and pyrrhotite in calcite; and in a specimen of brecciated vein material with chalcopyrite, pyrite, and tellurbismuth usually in

contact with galena; from the Porcupine Reef Gold Mines Ltd., Pamour, in several specimens of milky quartz with some greenish-white talc containing much visible gold, sometimes in subhedral crystals and grains, rarely in contact with minute areas of tellurbismuth and non-magnetic pyrrhotite which has an unusual deep brownish-bronze colour; in Quebec, from the Lamaque Mine, Bourlamaque Township, in a specimen of white quartz with small plates of tellurbismuth and massive calaverite; from the Louvicourt Goldfields Mine, Louvicourt Township, in several specimens of tourmalinized quartz with much free gold and small areas of calaverite and tarnished tellurbismuth in close association with calcite; from the Robb-Montbray Mine, Montbray Township, in a dozen pieces of solid rich aggregates of tellurides, sulphides, and gold with substantial masses of tellurbismuth and altaite; from the Horne Mine, Noranda, in a polished section with tellurbismuth intergrown in part with altaite; and from the Sullivan Consolidated Mines, disseminated with petzite in massive white quartz.

<i>Analyses</i>	1	2	A
Bi.....	49.7	47.7	52.2
Te.....	45.1	47.9	47.8
S.....	none	none	—
	94.8	95.6	100.0

1. Hunter Mine, Khutze Inlet, near Swanson Bay, Skeena Mining Division, British Columbia. Anal. Forward (in Warren & Davis, 1940, p. 110).

2. Ashloo Mine, near Squamish, Howe Sound, Vancouver Mining Division, British Columbia. Anal. Forward (in Warren & Davis, 1940, p. 110).

A. Calculated for  $\text{Be}_2\text{Te}_3$ .

#### Wehrlite— $\text{Bi}_{2+x}\text{Te}_{3-x}$

Hexagonal,  $R\bar{3}m$ ;  $a = 4.42$ ,  $c = 29.85$ ;  $r = 10.27$ ,  $\alpha = 24^\circ 51'$ ;  $Z = 1$  (Peacock, in Warren & Peacock, 1945).

In foliated masses, massive to finely granular; colour tin-white to steel-gray. Once observed with a peculiar bronzy tarnish. Perfect cleavage giving flexible and slightly elastic folia;  $H = 2$  (B);  $G = 8.37\text{--}8.44$ .

Polished sections white with weak anisotropism (light to dark gray).  $\text{HNO}_3$  stains black;  $\text{HCl}$  stains light gray-brown;  $\text{KCN}$  negative;  $\text{FeCl}_3$  stains iridescent to purplish black;  $\text{KOH}$ ,  $\text{HgCl}_2$  negative.

Wehrlite may best be considered as a variety of tellurbismuth. It is essentially a solid solution of Bi in  $\text{Bi}_2\text{Te}_3$  with Bi approximately at the saturation limit (about 60 wt. per cent Bi) found in the artificial system (Peacock, 1945, p. 68), giving a composition near  $\text{BiTe}$ .

*Occurrences*: Deutsch-Pilsen (Hungary). In Canada this rare mineral has been positively identified in British Columbia, Ontario, and Quebec.

*X-Ray Powder Pattern (R.M.T.; fig. 19)*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
2	9.0	4.91	3	25.0	1.819	1	42.4	1.140
1	11.6	3.82	3	28.6	1.606	1	44.1	1.105
1	12.4 $\beta$	3.23	3	31.2	1.484	2	45.65	1.075
10	13.8	3.22	4	33.25	1.402	2	47.6	1.041
7	19.0	2.36	2	36.2	1.301	1	50.3	0.999
5	20.4	2.21	1	37.05	1.275			
3	22.7	1.992	2	40.65	1.180			

In British Columbia, at the White Elephant Claim, Okanagan Lake, Vernon Mining Division, in several specimens of white quartz containing a soft massive mineral with a bronzy tarnish (wehrlite) and a soft platy mineral (tetradymite); from the Marble Bay and Little Billie Mines, Texada Island, Nanaimo Mining Division, as several polished sections with wehrlite and hessite in minute amounts, in close association with bornite and chalcopyrite; from the Charlie Vein, Tchaikazan River, Taseko Lake district, as minute amounts in polished sections, associated with galena and hessite; in Ontario from Painkiller Lake, Beatty Township, as a polished section with minute amounts of wehrlite and gold; from the Treadwell property, Painkiller Lake, Beatty Township, as several specimens of white quartz with disseminated wehrlite; in Quebec, from the Canadian Malartic Gold Mines Ltd., Fourniere Township, as thin films on rather glassy quartz, associated with grains of petzite, gold, and pyrite, and at the Bevcourt Mine, Louvicourt Township, disseminated with altaite, petzite, and gold in massive white quartz.

Although there are no analyses of Canadian wehrlite, the occurrence of this mineral in close association with silver-rich minerals like hessite and petzite suggests that the small silver content of the type Hungarian material is combined in included silver minerals.

**Tetradymite**— $\text{Bi}_2\text{Te}_2\text{S}$ 

Hexagonal,  $R\bar{3}m$ ;  $a=4.21$ ,  $c=29.43$ ;  $r=10.11$ ,  $\alpha=24^\circ 2\frac{1}{2}'$ ;  $Z=1$  (Peacock)

Commonly as foliated to bladed masses; rarely as acutely rhombohedral crystals; twins (fourlings) are reported. Steel-gray with metallic splendid lustre, tarnishing dull gray, black, or iridescent. Perfect basal cleavage yielding flexible but not elastic laminae;  $H=1\frac{1}{2}$  (B);  $G=7.3 \pm 0.2$ .

Polished sections bright white with weak anisotropism, polarization colours light to dark gray.  $\text{HNO}_3$  stains through iridescent to black;  $\text{HCl}$  negative (2 minutes);  $\text{KCN}$  negative;  $\text{FeCl}_3$  stains iridescent;  $\text{KOH}$ ,  $\text{HgCl}_2$  negative.



*X-Ray Powder Pattern (R.M.T.; fig. 20)*

<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$	<i>I</i>	$\theta(\text{Cu})$	$d(\text{meas.})$
1	8.95	4.94	$\frac{1}{2}$	25.8	1.766	$\frac{1}{2}$	40.6	1.181
$\frac{1}{2}$	12.2	3.64	3	26.1	1.747	$\frac{1}{2}$	45.4	1.080
$\frac{1}{2}$	13.75	3.24	4	27.95	1.640	1	47.0	1.051
10	14.35	3.10	1	18.7	1.601	3	49.45	1.012
1	16.2	2.76	1	29.7	1.551	1	51.7	0.979
2	17.25	2.59	$\frac{1}{2}$	30.7	1.506	1	53.0	0.962
2	18.25	2.45	2	32.3	1.439	2	54.8	0.941
4	19.7	2.28	1	32.7	1.423	2	57.25	0.914
2	20.8	2.16	2	34.8	1.347	3	59.7	0.980
3	21.4	2.11	4	36.5	1.292	3	66.3	0.839
3	23.05	1.963	2	37.8	1.254	3	69.0	0.823
3	23.65	1.916	$\frac{1}{2}$	39.2	1.216			
$\frac{1}{2}$	25.0	1.819	4	39.55	1.207			

*Occurrences:* Hungary, Romania, Sweden, Norway, Japan, Rhodesia, Queensland, New South Wales, Western Australia, Bolivia, New Mexico, Arizona, California, Colorado, Idaho, Montana, Virginia, North Carolina, South Carolina, Georgia. Observed from Boliden, Sweden, as a coarsely crystalline compact mass (40×30 mm.) of tellurbismuth and tetradymite; at the Red Cloud Mine, Boulder County, Colorado, as a compact mass of petzite, tetradymite, and pyrite.

In Canada, the occurrence of tetradymite has been verified at the following localities: in the Yukon Territory, at Dublin Gulch, Mayo district, as a small placer pebble with an embedded subhedral crystal of gold; at the Selwyn River, as a few small cleavage flakes; at Discovery Fork, and the East Fork of Nansen Creek, Carmacks district, as several placer pebbles up to 10 mm. in length; in British Columbia at the Harrison Group, Lindquist Lake, Omineca Mining Division, in a polished section, and in several pieces of vein quartz with flakes of tetradymite; at the White Elephant (Precambrian) Mine, Okanagan Lake, Vernon Mining Division, as several specimens of quartz with flakes of tetradymite; at the Jumbo Mine, Rossland, as a thin film of tetradymite and gold on monzonite; at the Taylor Windfall Mine, Clinton Mining Division; in Ontario, at the Broulan Mine, Porcupine district, as a 3 mm. band with minor gold in massive white quartz; at the McKellar Longworth Mine, Schreiber, in a polished section with chalcopyrite, and gold; and at the McKenzie Red Lake Gold Mines, in a polished section with galena; in Quebec, at the Lamaque Mine, Broulamaque Township, as a specimen of white quartz with small cleavage flakes of tetradymite; at the Perron Mine, Broulamaque, as several pieces of coarsely crystalline calcite with

films of bronzy tetradymite associated with gold; at the Siscoe Gold Mines, Siscoe, as a specimen of tourmalinized quartz with small areas of calcite with coarse gold rarely in contact with 1 mm. grains of tetradymite; and at the Powell Rouyn Mine, Noranda, as a somewhat decomposed stringer of tetradymite in a dark green pyritized quartzose rock coated on two sides by greenish-black chlorite, pink calcite crystals, pyrite, and free gold.

In addition, tetradymite has been reported from British Columbia, Ontario, and Quebec. In British Columbia, tetradymite was reported from near Liddle Creek, Kaslo River, West Kootenay district (Hoffmann, 1897, p. 10R); the Rhoderic Dhu Claim, Long Lake Camp, Greenwood Mining Division (Kemp, 1898, p. 317); the Nickel Plate Mine, near Hedley, Osoyoos Mining Division (Camsell, 1910, p. 138); Glacier Gulch near Smithers, Omineca Mining Division (Pratt, 1931, p. 56); in Ontario from the Howey Gold Mine, Red Lake (Cornford, in Horwood, 1945, p. 144); from the Toburn Mine, Kirkland Lake (Campbell & Deyell, in Hopkins, 1915, p. 183); the Mikado Mine, Rickard Township (Hopkins, 1915, p. 183); the Hattie, Treadwell, Mayo, Hull, and Cartwright properties near Painkiller Lake, Beatty Township, and the Miller Independence Mine, Boston Creek, Pacaud Township (Hopkins, 1921, pp. 9, 21; this material is tellurbismuth according to Berry—*priv. comm.*); Straw Lake near Fort Frances (Thomson, 1934, p. 33); Bigstone Bay, Lake of the Woods, and the Gold Shore Mine, Red Lake (Thomson, 1935, p. 48); the Hollinger Mine, Porcupine district (Keys, 1940, p. 620); the Chambers-Ferland Group, Schreiber (Thomson, 1923, p. 39); in Quebec, from the

<i>Analyses</i>	1	2	3	4	5	A
Bi.....	51.85	59.10	60.72	60.88	61.05	59.27
Pb.....	3.50	—	—	—	—	—
Ag.....	0.91	—	—	—	—	—
S.....	4.30	4.85	4.29	4.29	3.65	4.54
Te.....	36.01	35.90	34.71	34.47	35.10	36.19
Insol.....	3.52	—	—	—	—	—
	100.09	99.85	99.72	99.64	99.80	100.00

1. Liddle Creek, Kaslo River, West Kootenay district, British Columbia. Tetradymite with minor altaite and hessite; incl. Se, Tl, trace. Anal. Johnston (in Hoffmann, 1897, p. 10R).

2. White Elephant Claim, Okanagan Lake, Vernon Mining Division, British Columbia. Anal. Forward (in Warren, 1946, p. 77).

3, 4. White Elephant Claim, Okanagan Lake, Vernon Mining Division, British Columbia; incl. Se trace. Anal. Williams (in Warren, 1946, p. 77).

5. White Elephant Claim, Okanagan Lake, Vernon Mining Division, British Columbia (tetradymite with minor wehrlite); incl. Se trace. Anal. Williams (in Warren, 1946, p. 77).

A. Calculated for  $\text{Bi}_2\text{Te}_2\text{S}$ .

Eureka Mine, Tiblemont Township (Thomson, 1934, p. 35); the Horne Mine, Noranda (Price, 1934, p. 132); and the McWatters Mine, Rouyn Township (Thomson, 1935, p. 48). Some of these reported occurrences may refer in fact to other bismuth tellurides.

#### *Uncertain or Discredited Tellurides*

##### STUETZITE— $\text{Ag}_4\text{Te}$ (?)

A supposed specimen of the uncertain silver telluride stuetzite (USNM, R 442, from Siebenbürgen, Transylvania), was obtained through the kindness of Dr. E. P. Henderson of the United States National Museum. This material gave the  $x$ -ray powder pattern of bournonite. An attempt to make the compound  $\text{Ag}_4\text{Te}$  by pyrosynthesis resulted in the formation of hessite,  $\text{Ag}_2\text{Te}$ , and excess silver.

##### MUTHMANNITE— $(\text{Ag}, \text{Au})\text{Te}$

No specimen of this mineral was available for study. Analyses have indicated the composition  $(\text{Ag}, \text{Au})\text{Te}$ , but fusions of this composition, with  $\text{Ag}:\text{Au}=1:1$  and  $\text{Ag}:\text{Au}=2:1$ , gave mixtures of calaverite,  $\text{AuTe}_2$ , and petzite,  $\text{Ag}_3\text{AuTe}_2$ . The specific gravity 5.598 given by Scharizer (1880, p. 605) for muthmannite is apparently too low since it is much lower than that of each of the components.

##### NIGGLITE— $\text{PtTe}_3$ (?)

This is the name given by Scholtz (1936, p. 184) to a mineral believed to be a platinum telluride, which gave 34.8 per cent Pt on analysis of a single grain weighing a fraction of a milligram. No specimen was available. An attempt to produce  $\text{PtTe}_3$  by pyrosynthesis gave  $\text{PtTe}_2$  (not niggliite) and excess Te.

##### "ANTAMOKITE"

"Antamokite" was supposed to be a new telluride of gold and silver. A recent study of ore from the type locality, the Benguet Consolidated Mining Company Mine, Antamok, Mountain Province, Philippine Islands (Thompson, 1946 *b*), indicated that this material is petzite associated with calaverite.

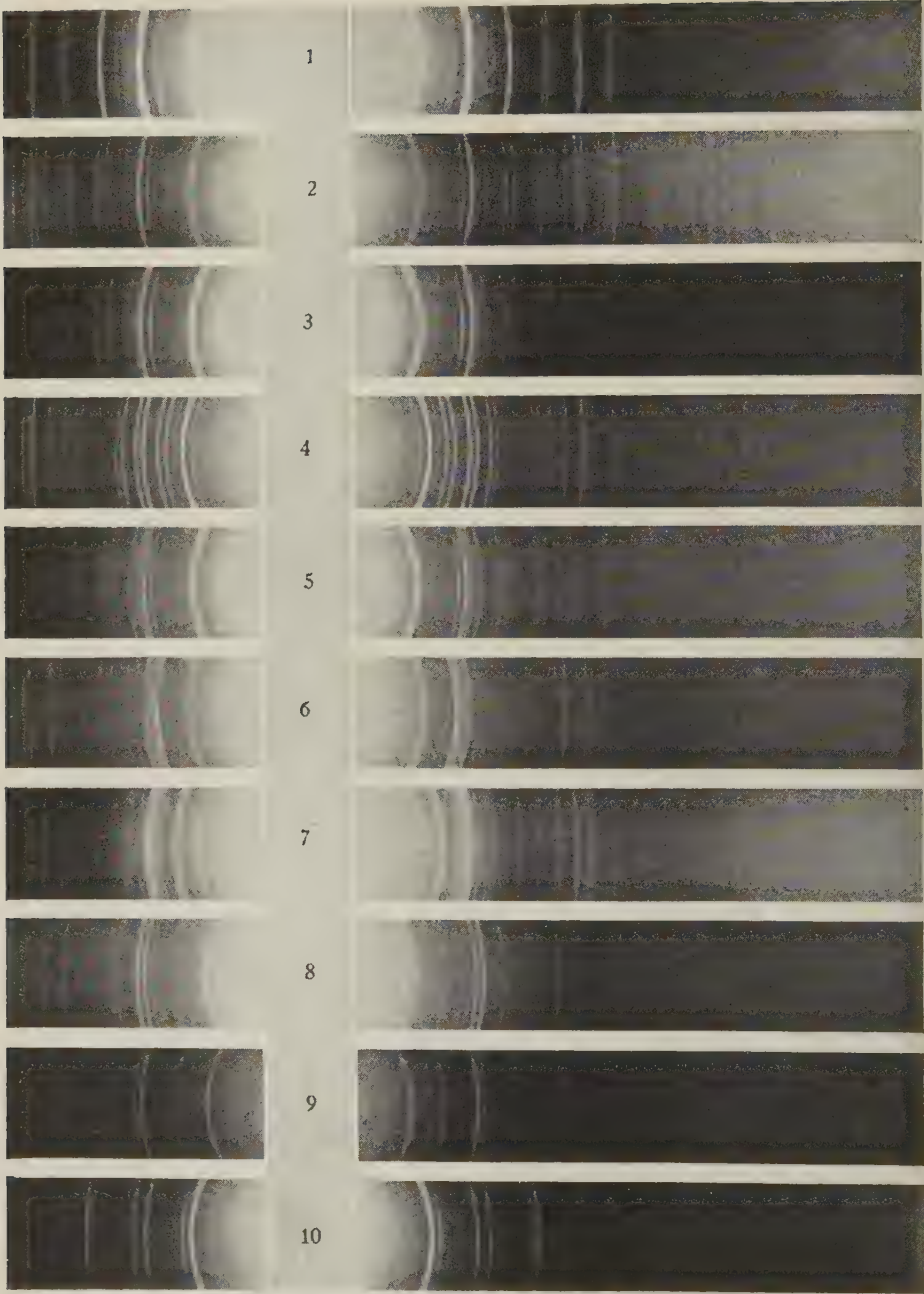
##### "GOLDFIELDITE"

This dubious compound of Cu, Sb, Te, and S was recently shown (Thompson, 1946*a*) to be a previously unknown tellurian variety of tetrahedrite, with  $a=10.35$ .

#### X-RAY POWDER PHOTOGRAPHS

The accompanying figures are actual size reproductions of contact prints from  $x$ -ray powder films taken in a camera of radius  $360/4\pi$  mm.





( $1^\circ \theta = 1$  mm. on film). Cu radiation (Ni filter) was used in all cases except Fig. 11, for which Fe radiation (Mn filter) was used.

## CANADIAN TELLURIDE LOCALITIES

In conclusion it will be useful to give a list of the known Canadian localities for telluride minerals, with the names of the minerals reported at each locality. This list combines published information, derived mainly from the works of Cairnes, Todd, and Ellis Thomson, with the results of the present work. Mineral names marked \* are positive identifications based on *x*-ray powder photographs, while names in quotation marks are proved misnomers. It is hoped that this improved list will be useful to prospectors and geologists as well as providing reliable additions to the data of topographic mineralogy.

*Yukon Territory*

Upper Burwash Creek, Kluane District.....	*Altaite
	*Hessite
	*Tellurbismuth
	*Hedleyite
Dublin Gulch, Mayo District.....	*Tetradymite
Selwyn River, Selkirk District.....	*Tetradymite
East Fork and Discovery Fork of Nansen Creek, Carmacks District....	*Tetradymite
Buffalo Hump Group, Mt. Stevens, Wheaton River District.....	*Hessite
	Sylvanite
Gold Reef Claim, Wheaton River District.....	Sylvanite
	Hessite
	Petzite

*British Columbia*

Engineer Mines, Taku Arm, Tagish Lake, Atlin Mining Division.....	Calaverite
Harrison Group, Lindquist Lake, Omineca Mining Division.....	*Hessite
	*Tetradymite
Glacier Gulch, Hudson Bay Mountain, Omineca Mining Division.....	*Joseite A, B
	("tetradymite")
	Calaverite
Hunter Group Claims, Khutze Inlet, Skeena Mining Division.....	*Tellurbismuth

FIG. 1. Montbrayite, Robb-Montbray Mine, Montbray Township, Quebec.

FIG. 2. Calaverite, Cripple Creek, Colorado.

FIG. 3. Krennerite, Cripple Creek, Colorado.

FIG. 4. Petzite, Kalgoorlie, Western Australia.

FIG. 5. Sylvanite, Nagyág, Transylvania.

FIG. 6. Hessite, Botés, Transylvania.

FIG. 7. Empressite, Red Cloud Mine, Boulder County, Colorado.

FIG. 8. Weissite, Good Hope Mine, Vulcan, Colorado.

FIG. 9. Rickardite, Good Hope Mine, Vulcan, Colorado.

FIG. 10. Melonite, Robb-Montbray Mine, Montbray Township, Quebec.

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Charlie Group, Taseko Lake, Clinton Mining Division.....	*Altaite *Hessite *Wehrlite
Hido Group (Pellaire Gold Mines property), Clinton Mining Division...	*Altaite *Hessite
Taylor Windfall Mine, Clinton Mining Division.....	*Tetradymite
Windpass Mine, Chu Chua, Kamloops Mining Division.....	*Joseite A
Valdez Island, Nanaimo Mining Division.....	Gold tellurides
Marble Bay Mine, Texada Island, Nanaimo Mining Division.....	*Hessite *Wehrlite
Little Billie Mine, Texada Island, Nanaimo Mining Division.....	*Hessite *Wehrlite
Ashloo Mine, near Squamish, Howe Sound, Vancouver Mining Division.	*Tellurbismuth
Nickel Plate Mine, near Hedley, Osoyoos Mining Division.....	Tetradymite
Good Hope Claim, near Hedley, Osoyoos Mining Division.....	*Joseite B *Hedleyite
Hedley Monarch Mine, Olalla, Osoyoos Mining Division.....	*Altaite *Hessite *Pezite
Hedley Yuniman Gold Fields Limited, Bradshaw Creek, Osoyoos Mining Division.....	*Tellurbismuth
Calumet Claim, Kruger Mountain, Osoyoos Lake, Osoyoos Mining Division.....	Hessite Pezite
White Elephant Claim, Okanagan Lake, Vernon Mining Division.....	*Tetradymite *Wehrlite
Boundary District.....	*Hessite
Long Lake Camp, Greenwood Mining Division	
(a) Jewel Mine.....	Rich tellurides
(b) Lakeview Claims.....	*Altaite *Hessite
(c) North Star Claim.....	*Hessite
(d) Enterprise Claim.....	Pezite
(e) Rhoderic Dhu Claim.....	Altaite Tetradymite
Jumbo Mine, Rossland, Trail Creek Mining Division.....	*Tetradymite
Olive Mabel Claim, Gainor Creek, Lardeau Mining Division.....	Nagyagite

FIG. 11. Frobergite, Robb-Montbray Mine, Montbray Township, Quebec.

FIG. 12. Altaite, Lake Shore Mine, Kirkland Lake, Ontario.

FIG. 13. Nagyagite, Nagyág, Transylvania.

FIG. 14. Coloradoite, Toburn (Tough-Oakes) Mine, Kirkland Lake, Ontario.

FIG. 15. Hedleyite, Good Hope Claim, Near Hedley, Osoyoos Mining Division, British Columbia.

FIG. 16. Joseite A, Glacier Gulch, Hudson Bay Mountain, near Smithers, British Columbia.

FIG. 17. Joseite B, Good Hope Claim, near Hedley, Osoyoos Mining Division, British Columbia.

FIG. 18. Tellurbismuth, Hedley Yuniman Gold Fields Limited, Bradshaw Creek, Osoyoos Mining Division, British Columbia.

FIG. 19. Wehrlite, Treadwell property, near Painkiller Lake, Beatty Township, Ontario.

FIG. 20. Tetradymite, Selwyn River, Selkirk district, Yukon Territory.

Liddle Creek, Kaslo River, Ainsworth Mining Division.....	Altaite Hessite *Tetradymite
Pay Roll Mine, near Cranbrook, Fort Steele Mining Division.....	Altaite

*Manitoba*

Copper Lake.....	*Altaite
San Antonio Mine, near Bissett.....	*Petzite

*Ontario*

Ardeen (Huronian, Moss, Shebandowan) Mine, Moss Township.....	*Altaite *Hessite *Petzite *Tellurbismuth Coloradoite Nagyagite Sylvanite
Gold Creek, Pine Portage Bay, Lake of the Woods.....	Hessite
Three Ladies Mine, Lake of the Woods.....	Altaite
Bigstone Bay, Lake of the Woods.....	Hessite Petzite Calaverite Sylvanite Tetradymite
Gold Shore Mine, Red Lake.....	Tetradymite
Gold Eagle Mines, Red Lake.....	Altaite Sylvanite
Howey Gold Mine, Red Lake.....	Altaite Sylvanite Tetradymite
McKenzie Red Lake Gold Mines, Red Lake.....	Altaite Petzite Krennerite Tetradymite
Chambers-Ferland Group, near Schreiber.....	Altaite Tetradymite
McKellar Longworth Mine, Schreiber.....	*Tetradymite
Jackson-Manion Mine, District of Kenora.....	Petzite
Straw Lake, near Fort Frances.....	Tetradymite
Anderson Farm, Deloro Township.....	Petzite
Powell Claim, Deloro Township.....	*Hessite
Labine-Smith Claim, Maisonville Township.....	Petzite
Ashley Mine, Bannockburn Township.....	*Altaite Krennerite
Hattie, Hull, Cartwright, Mayo, and Treadwell properties, near Pain- killer Lake, Beatty Township.....	*Wehrlite ("tetradymite") Calaverite
Miller Independence and Boston McRae Mines, Boston Creek, Pacaud Township.....	*Calaverite *Tellurbismuth ("tetradymite")

Hollinger Mine, Porcupine District.....	*Coloradoite *Petzite *Hessite Tetradymite
Dome Mine, Porcupine District.....	*Altaite Calaverite Sylvanite
Broulan Mine, Porcupine District.....	*Tetradymite
Porcupine Reef Gold Mines Limited, Pamour.....	*Tellurbismuth
A prospect in Tisdale Township.....	*Hessite
Kirkland Lake Camp	
Kirkland Lake Mine.....	*Altaite *Coloradoite *Hessite
Teck-Hughes Mine.....	*Altaite Coloradoite Calaverite
Sylvanite Mine.....	*Altaite Petzite Coloradoite Calaverite
Toburn (Tough-Oakes) Mine.....	*Altaite *Coloradoite *Petzite *Calaverite *Melonite Hessite Tetradymite
Wright Hargreaves Mine.....	*Altaite *Coloradoite *Calaverite *Melonite Petzite
Lake Shore Mine.....	*Altaite *Calaverite *Coloradoite *Petzite
Macassa Mine.....	*Altaite *Calaverite *Melonite
Bidgood Mine.....	*Altaite *Coloradoite *Calaverite
Kirkland Golden Gate Mine, Swastika.....	*Altaite *Calaverite
Upper Canada Mine, Gauthier Township.....	*Altaite *Petzite *Calaverite
Mikado Mine, Rickard Township.....	Tetradymite



## Quebec

Opasatica District.....	Petzite
Robb-Montbray Mine, Montbray Township, Abitibi County.....	*Altaite
	Coloradoite
	*Petzite
	*Melonite
	*Frohbergite
	*Montbrayite
	("krennerite")
	*Tellurbismuth
	("tetradymite")
Horne Mine, Noranda.....	*Altaite
	*Calaverite
	*Petzite
	*Tellurbismuth
	("tetradymite")
	Hessite
	Krennerite
	Sylvanite
	Rickardite (?)
	Undetermined
	pink telluride
Powell Rouyn Mine, Noranda.....	*Tetradymite
Stadacona Mine, Rouyn.....	*Petzite
McWatters Mine, Rouyn Township.....	Hessite
	Tetradymite
Lamaque Mine, Bourlamaque Township.....	*Petzite
	*Calaverite
	*Tellurbismuth
	*Tetradymite
Perron Mine, Bourlamaque Township.....	*Tetradymite
Sullivan Mine, Bourlamaque Township.....	*Hessite
Sullivan Consolidated Mines.....	*Tellurbismuth
	*Petzite
Siscoe Gold Mines, Siscoe.....	*Tetradymite
Eureka Mine, Tiblémont Township.....	Tetradymite
Louvicoourt Goldfields Mine, Louvicoourt Township.....	*Calaverite
	*Tellurbismuth
Bevcourt Mine, Louvicoourt Township.....	*Altaite
	*Petzite
	*Wehrlite
Canadian Malartic Gold Mine, Fourniere Township.....	*Petzite
	*Calaverite
	*Wehrlite

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# OBSERVATIONS ON SOME ALUMINIUM FLUORIDE MINERALS<sup>1</sup>

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## ABSTRACT

Jarlite ( $G=3.87$ ) is monoclinic, tabular on (001); space-group  $C2/m$ ,  $C2$ , or  $Cm$ ;  $a=15.99$ ,  $b=10.82$ ,  $c=7.24$  kX,  $\beta=101^\circ 49'$ ;  $Y=b$ ,  $Z:c=+(84^\circ \pm 2^\circ)$ ;  $\alpha=1.430$ ,  $\beta=1.435$ ,  $\gamma=1.437$ ,  $2V=90^\circ \pm 10^\circ$ ; the composition  $NaSr_3Al_3F_{16}$  is apparently in error. "Metajarlite" ( $G=3.65$ , with inclusions), with  $\alpha=1.429$ ,  $\beta=1.431$ ,  $\gamma=1.434$ , and an  $x$ -ray powder pattern very like that of jarlite, is merely a variety of jarlite. Prosopite is monoclinic,  $C2/c$ ;  $a=6.67$ ,  $b=11.17$ ,  $c=7.37$  kX,  $\beta=94^\circ 56'$ . Weberite is orthorhombic,  $Ibmm$  or  $Ibm2$ ; the unit cell with  $a=7.30$ ,  $b=7.05$ ,  $c=9.97$  kX, contains  $4[Na_2MgAlF_7]$ ;  $X=a$ ,  $Y=b$ . Cryolithionite has  $a=12.14$  kX. Powder photographs of ralstonite show indications of non-cubic symmetry, and "hagemannite" is an ochre consisting of ralstonite with some thomsenolite and goethite.  $X$ -ray powder spacings and intensities (10 scale) are given for gearksutite and ralstonite, and similar data with indices and calculated spacings are given for cryolite, jarlite and "metajarlite," prosopite, and weberite. All the material studied, except prosopite from Colorado, came from Ivigtut, Greenland.

Occurring with the cryolite at the most famous locality, Ivigtut, Greenland, are representatives of most of the rare aluminium fluoride minerals. An excellent suite of specimens from the Ivigtut quarry was kindly made available to the author by Professor H. S. Scott of the Department of Geology, McMaster University, Hamilton, Ontario. Professor Scott had collected these specimens at Ivigtut in 1942 when, as geologist for Mr. Hans Lundberg of Toronto, he made a geological examination of the cryolite deposit. A crystallographic investigation of the lesser known aluminium fluoride minerals in this suite was carried out in the Mineralogical Laboratory of the Department of Geological Sciences, University of Toronto, under the helpful and stimulating direction of Professor M. A. Peacock. Some of the results of this investigation, those on thomsenolite and pachnolite, have already been published (Ferguson, 1946); the present paper summarizes the new and confirmatory observations on the remaining minerals. The essence of this paper was presented before the Mineralogical Society of America at the 1947 meeting in Ottawa and has been published in abstract (Ferguson, 1948).

Detailed studies were made of jarlite  $NaSr_3Al_3F_{16}(?)$  and weberite  $Na_2MgAlF_7$  as well as thomsenolite and pachnolite, both  $NaCaAlF_6 \cdot H_2O$ ; a small amount of work, mainly indexing the  $x$ -ray powder photographs, was carried out on the remaining minerals in the group (excluding

<sup>1</sup> Extracted from an unpublished Ph.D. thesis: A contribution to the mineralogy of the aluminium fluorides of Greenland—*University of Toronto, 1947*.

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elpasolite  $K_2NaAlF_6$  which has not been reported from Ivigtut): cryolite  $Na_3AlF_6$ , chiolite  $Na_5Al_3F_{14}$ , cryolithionite  $Na_3Li_3Al_2F_{12}$ , gearnakutite  $CaAl(F,OH)_5 \cdot H_2O$ , prosopite  $CaAl_2(F,OH)_8$  (from Colorado), ralstonite  $Na_{2-x}(Al,Mg)_2(F,OH)_6 \cdot 1-yH_2O$ .

All x-ray diffraction photographs were taken with Cu radiation, a Ni filter being used for the powder pictures. Results are given in kX units ( $CuK\alpha_1 = 1.5374$  kX). Intensities were visually estimated on a scale of 10.

#### CRYOLITE— $Na_3AlF_6$

An x-ray powder photograph of this mineral indexed well on the cell dimensions of Menzer (1928). The powder data indexed to  $\theta(Cu) = 29.35^\circ$  appear in Table 1, and agree well with the only previously published data (unindexed) in the ASTM Card Index (1943, card 3469).

#### CRYOLITHIONITE— $Na_3Li_3Al_2F_{12}$

Menzer (1930) has published powder data for this cubic mineral indexed on a cell edge of 12.097 Å. A new powder photograph gave slightly

TABLE 1. CRYOLITE— $Na_3AlF_6$ : X-RAY POWDER PATTERN  
Monoclinic,  $P2_1/n$ ;  $a = 5.39$ ,  $b = 5.59$ ,  $c = 7.76$  kX,  $\beta = 90^\circ 11'$ ;  $Z = 2$

<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)	<i>hkl</i>	<i>d</i> (calc.)	<i>I</i>	$\theta(Cu)$	<i>d</i> (meas.)	<i>hkl</i>	<i>d</i> (calc.)
7	9.9°	4.47	{(011) (101) (101)}	4.536 4.427	4	21.65°	2.08	{(122) (122) (212) (212)}	2.090 2.058
7	11.45	3.87	{(110) (002)	3.880 3.880	10	23.35	1.939	{(220) (004) (023)	1.940 1.940 1.898
2	12.85	3.46	{(111) (111)	3.470	1	24.0	1.890	{(221) (221)	1.882
9	16.25	2.75	{(020) (112) (112)	2.795 2.744	$\frac{1}{2}$	25.5	1.786	{(123) (123)	1.791
$\frac{1}{2}$	18.25	2.47	(200)	2.695	1	25.9	1.760	{(130) (130)	1.761
3	18.5	2.42	(120)	2.481	1	26.35	1.732	{(114) (114) (222) (222)	1.735 1.735 1.735
8	19.3	2.33	(210)	2.428	4	26.7	1.711	{(131) (113) (310)	1.717 1.711
			{(013) (103) (103)	2.348 2.332	2	27.4	1.670	{(311) (311)	1.670
			{(211) (211)	2.317	6	28.8	1.596	(024)	1.594
4	19.85	2.26	(022)	2.268	8	29.35	1.568	{(312) (312)}	1.565
1	20.35	2.21	{(202) (202)	2.213					
4	21.0	2.14	{(113) (113)}	2.152					

TABLE 1—*continued*

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
$\frac{1}{2}$	30.75°	1.503	4	38.8°	1.227	2	48.8°	1.022
$\frac{1}{2}$	31.05	1.490	$\frac{1}{2}$	39.35	1.212	1	49.75	1.007
1	31.9	1.455	1	41.45	1.161	$\frac{1}{2}$	52.65	0.967
1	32.6	1.427	$\frac{1}{2}$	42.7	1.134	$\frac{1}{2}$	53.85	0.952
1	33.4	1.396	$\frac{1}{2}$	43.35	1.120	$\frac{1}{2}$	54.7	0.942
4	34.05	1.373	$\frac{1}{2}$	44.0	1.107	$\frac{1}{2}$	56.5	0.922
1	34.7	1.350	2	45.2	1.083	2	57.1	0.916
2	35.75	1.316	$\frac{1}{2}$	46.45	1.061	2	57.9	0.907
1	36.1	1.305	$\frac{1}{2}$	47.0	1.051	1	59.4	0.893
$\frac{1}{2}$	37.1	1.274	2	47.45	1.043	1	62.55	0.866
$\frac{1}{2}$	38.25	1.242	2	48.15	1.032			

larger spacings than Menzer's and led to a cell edge of 12.14 kX. Except for the small differences in spacing, the new indexed powder data agreed well with Menzer's.

#### GEARKSUTITE— $\text{CaAl}(\text{F}, \text{OH})_5 \cdot \text{H}_2\text{O}$

This mineral occurs as white, earthy, kaolin-like masses at Ivigtut with thomsenolite and cryolite, and at several other localities in different association. Since macroscopic crystals are unknown, gearksutite does not permit of a single-crystal investigation which is necessary to interpret the complex powder pattern. Specimens of this mineral from Ivigtut and

TABLE 2. GEARKSUTITE— $\text{CaAl}(\text{F}, \text{OH})_5 \cdot \text{H}_2\text{O}$ : X-RAY POWDER PATTERN

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
1	6.6°	6.69	1	21.4°	2.11	3	31.85°	1.457
2	8.45	5.23	4	21.75	2.07	2	32.4	1.435
10	9.75	4.54	$\frac{1}{2}$	22.5	2.01	1	33.5	1.393
1	11.75	3.77	$\frac{1}{2}$	23.0	1.967	4	34.3	1.364
3	12.6	3.52	8	23.55	1.924	$\frac{1}{2}$	35.9	1.311 <i>b</i>
7	13.34	3.33 <i>d</i>	3	23.85	1.901	2	37.35	1.267
8	14.15	3.14	3	25.0	1.819	3	38.45	1.236
1	16.45	2.71	6	26.2	1.741	1	38.85	1.225
1	17.0	2.63	1	26.85	1.702	1	39.6	1.206
$\frac{1}{2}$	18.2	2.46	3	27.4	1.670	1	40.5	1.184
$\frac{1}{2}$	19.0	2.36	$\frac{1}{2}$	28.5	1.611	$\frac{1}{2}$	42.35	1.141 <i>b</i>
8	19.7	2.28	1	28.85	1.593	1	43.05	1.126
1	20.1	2.24	$\frac{1}{2}$	30.15	1.531	2	44.35	1.100
6	20.9	2.15	2	31.1	1.488			

*d* doublet.

*b* broad.

from the Chancellor Mine, Boulder County, Colorado, gave identical x-ray powder patterns. The observed intensities, glancing angles, and measured spacings for these appear in Table 2.

#### JARLITE— $\text{NaSr}_3\text{Al}_3\text{F}_{16}$ (?)

Jarlite, first described by Bøggvad (1933), occurs as colorless, flat crystals about 1 mm. in size, often fused in the shape of a fan and growing with thomsenolite in vugs in the cryolite. In the same paper Bøggvad described another new mineral occurring as grayish fan-shaped masses intergrown with chiolite. The two new minerals were found to have fairly similar optical properties and specific gravities, and chemical analyses of both, by Blix (in Bøggvad, 1933), led to the same ideal formula,  $\text{NaSr}_3\text{Al}_3\text{F}_{16}$ . Bøggvad considered the second mineral sufficiently different from jarlite to warrant a separate name, meta-jarlite.

In the present investigation, single-crystal photographs were taken of jarlite, and the powder photographs, optical constants, and specific gravities of both minerals were re-examined in order to clarify the relationship between them.

*X-Ray Crystallography.* From goniometric observations on crystals of typically poor development, Bøggvad showed that jarlite is monoclinic with tabular development parallel to the symmetry axis (*Y* optic direction) and a tendency towards elongation along this axis. Bøggvad found several faces besides the two opposite ones giving the tabular development, but the author could find only the latter on nearly a dozen crystals examined. For this reason, single-crystal orientation about the symmetry axis was possible only by adjusting in one plane, that normal to the faces, by the usual goniometric method, and in another plane, that containing the faces, by using the optical extinction direction in that plane. This method was applied with success to one jarlite crystal of which rotation, zero- and first-layer Weissenberg photographs were taken with unfiltered Cu radiation. These led to the possible space-groups  $C2/m$ ,  $C2$ ,  $Cm$  and, with the tabular faces basal, the cell dimensions,  $a=15.99$ ,  $b=10.82$ ,  $c=7.24$ , all  $\pm 0.02$  kX;  $\beta=101^\circ 49' \pm 20'$ . Only after this work had been completed was it discovered that the cell dimensions of jarlite had been determined earlier by Brosset (1942) who obtained  $a=15.99$ ,  $b=10.89$ ,  $c=7.25$  Å,  $\beta=78.2^\circ$  ( $101.8^\circ$ ). The later independently determined values amply confirm the accuracy of Brosset's cell dimensions. The x-ray powder data for jarlite indexed to  $\theta(\text{Cu})=25.1^\circ$  on the new cell dimensions are given in Table 3.

Since meta-jarlite lacks crystal form, no attempt was made to examine it by single-crystal x-ray photographs; however, a powder photograph was taken and the observed data for it are given in comparison with those



for jarlite in Table 3. Brosset (1942) published no powder data for the two minerals, but he does note that the two give generally similar powder patterns with line positions somewhat unlike but intensities largely in agreement. From this he concludes that the two minerals are likely isomorphous. The comparison in Table 3 verifies Brosset's observation that the two patterns are generally similar; spacings are slightly but consistently smaller for meta-jarlite, and there are some marked discrepancies in the intensities of corresponding lines. The differences, however, are not sufficiently great to alter Brosset's conclusion that the two minerals are probably isomorphous and indeed, meta-jarlite should probably be considered simply a variety of jarlite. This conclusion is strengthened by the similarity in other physical properties, notably the optical constants, of the two minerals as described below.

*Geometrical Crystallography.* An attempt was made to relate Bøggvad's morphological setting to the structural cell. Choosing the  $Y$  optic direction as  $b[010]$  and the prominent tabular form as  $\{100\}$ , Bøggvad derived the following axial elements from the measurement of several faces on crystals of admittedly poor development:  $a:b:c=1.46:1:2.58$ ,  $\beta=69^\circ 20'$  ( $110^\circ 40'$ ). Since, as noted above, the tabular faces alone could be detected on the crystals used in the present investigation, the only direct evidence for relating the two settings is the coincidence of  $b[010]$  in both (from the optics), and the equivalence of morphological  $\{100\}$  with structural  $\{001\}$ . With this limited information, the only method of relating the two settings completely is to derive, within the prescribed limitations, all reasonable transformations, calculate some of Bøggvad's observed interfacial angles for each transformation using the  $x$ -ray elements, and judge from the agreement between calculated and observed values which transformation is most probable. With this procedure, several possible transformations were derived of which the following appears to be the most likely:

Bøggvad to Ferguson:  $00\bar{4}/060/301$

Applying this, Bøggvad's forms  $\{100\}$ ,  $\{010\}$ ,  $\{001\}$ ,  $\{110\}$ ,  $\{101\}$  become respectively  $\{001\}$ ,  $\{010\}$ ,  $\{401\}$ ,  $\{021\}$ ,  $\{201\}$  in the structural setting. Due to the bad development of the crystals, agreement between the transformed morphological elements and the  $x$ -ray elements is very poor:

	$a$		$b$		$c$		$\beta$
Goniometric (Bøggvad)	1.72	:	1	:	0.704		$104^\circ 10'$
$X$ -ray (Ferguson)	1.478	:	1	:	0.669		$101^\circ 49'$

Following is a comparison of Bøggvad's measured angles with those calculated from the new  $x$ -ray elements using the above transformation:

TABLE 3. JARLITE AND META-JARLITE: X-RAY POWDER PATTERNS

Jarlite Monoclinic, $C2/m$ $a=15.99$ , $b=10.82$ , $c=7.24$ kX, $\beta=101^\circ 49'$					Meta-Jarlite		
$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$
1	$7.45^\circ$	5.93	(201)	5.888	2	$7.55^\circ$	5.85
—	—	—	—	—	$\frac{1}{2}$	9.45	4.68
$\frac{1}{2}$	10.2	4.34	(021)	4.301	1	10.3	4.30
$\frac{1}{2}$	11.25	3.94	(400)	3.913	$\frac{1}{2}$	11.5	3.86
4	12.25	3.62	(311)	3.621	6	12.35	3.59
4	12.9	3.44	( $\bar{1}$ 12)	3.434	6	12.85	3.46
9	14.0	3.18	$\begin{Bmatrix} (420) \\ (401) \end{Bmatrix}$	$\begin{Bmatrix} 3.170 \\ 3.613 \end{Bmatrix}$	9	14.2	3.13
6	14.35	3.10	$\begin{Bmatrix} (\bar{3}12) \\ (\bar{4}21) \end{Bmatrix}$	$\begin{Bmatrix} 3.120 \\ 3.092 \end{Bmatrix}$	7	14.5	3.07
10	15.0	2.97	$\begin{Bmatrix} (202) \\ (\bar{5}11) \\ (022) \end{Bmatrix}$	$\begin{Bmatrix} 3.010 \\ 2.987 \\ 2.965 \end{Bmatrix}$	10	15.1	2.95
$\frac{1}{2}$	15.6	2.86	( $\bar{3}$ 31)	2.858	1	15.65	2.85
$\frac{1}{2}$	16.5	2.71	$\begin{Bmatrix} (421) \\ (040) \end{Bmatrix}$	$\begin{Bmatrix} 2.730 \\ 2.705 \end{Bmatrix}$	2	16.6	2.69
$\frac{1}{2}$	17.0	2.63	$\begin{Bmatrix} (\bar{6}01) \\ (222) \end{Bmatrix}$	$\begin{Bmatrix} 2.629 \\ 2.628 \end{Bmatrix}$	1	17.1	2.61
—	—	—	—	—	1	17.75	2.52
3	19.05	2.36	$\begin{Bmatrix} (\bar{6}21) \\ (530) \\ (003) \\ (\bar{1}13) \end{Bmatrix}$	$\begin{Bmatrix} 2.364 \\ 2.364 \\ 2.363 \\ 2.353 \end{Bmatrix}$	2	$19.1^\circ$	2.35
—	—	—	—	—	1	19.3	2.33
3	20.2	2.23	$\begin{Bmatrix} (\bar{4}03) \\ (440) \end{Bmatrix}$	$\begin{Bmatrix} 2.236 \\ 2.225 \end{Bmatrix}$	1	20.25	2.22
3	20.4	2.21	(113)	2.221	5	20.5	2.20
7	20.9	2.15	$\begin{Bmatrix} (023) \\ (332) \\ (\bar{6}22) \\ (203) \end{Bmatrix}$	$\begin{Bmatrix} 2.166 \\ 2.155 \\ 2.150 \\ 2.144 \end{Bmatrix}$	8	21.05	2.14

TABLE 3—(continued)

Jarlite					Meta-Jarlite		
<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	( <i>hkl</i> )	<i>d</i> (calc.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
$\frac{1}{2}$	21.9	2.06	$\begin{cases} (\overline{4}23) \\ (\overline{5}13) \end{cases}$	$\begin{matrix} 2.066 \\ 2.066 \end{matrix}$	—	—	—
4	22.7	1.992	$\begin{cases} (350) \\ (801) \\ (223) \\ (\overline{4}42) \\ (711) \end{cases}$	$\begin{matrix} 1.999 \\ 1.993 \\ 1.993 \\ 1.992 \\ 1.983 \end{matrix}$	1	22.7	1.992
2	23.0	1.967	$\begin{cases} (313) \\ (\overline{6}03) \end{cases}$	$\begin{matrix} 1.971 \\ 1.963 \end{matrix}$	7	23.05	1.963
—	—	—	—	—	1	23.5	1.928
—	—	—	—	—	1	24.25	1.872
1	24.35	1.864	(403)	1.861	2	24.45	1.857
6	25.1	1.812	$\begin{cases} (\overline{5}33) \\ (622) \\ (\overline{2}04) \end{cases}$	$\begin{matrix} 1.818 \\ 1.811 \\ 1.811 \end{matrix}$	2	25.05	1.816

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas)
—	—	—	4	25.35°	1.795
2	25.55°	1.782	2	25.7	1.773
—	—	—	$\frac{1}{2}$	26.5	1.723
1	26.85	1.702	3	27.2	1.682
—	—	—	$\frac{1}{2}$	28.3	1.621
2	28.9	1.591	1	29.0	1.586
$\frac{1}{2}$	29.4	1.566	$\frac{1}{2}$	29.45	1.564
—	—	—	$\frac{1}{2}$	29.9	1.542
2	30.65	1.508	3	30.65	1.508
1	31.4	1.475	1	31.65	1.465
$\frac{1}{2}$	32.4	1.435	1	32.6	1.427
$\frac{1}{2}$	33.35	1.398	1	33.5	1.393
$\frac{1}{2}$	33.9	1.378	1	34.75	1.349
2	34.9	1.344	$\frac{1}{2}$	35.0	1.340
1	35.25	1.332	$\frac{1}{2}$	35.7	1.317
$\frac{1}{2}$	36.3	1.299	$\frac{1}{2}$	36.35	1.297
—	—	—	$\frac{1}{2}$	36.9	1.280
$\frac{1}{2}$	37.3	1.269	1	37.45	1.264
—	—	—	$\frac{1}{2}$	38.15	1.244
1	39.45	1.210	$\frac{1}{2}$	40.0	1.196
1	40.75	1.178	3	40.75	1.178
$\frac{1}{2}$	45.45	1.079	—	—	—
$\frac{1}{2}$	46.85	1.054	—	—	—
$\frac{1}{2}$	50.3	0.999	1	51.4	0.984

Morph. (Bøgvad)	Structural	Meas. (Bøgvad)	Calc. (Ferguson)
(100):(010)	(001):(010)	90° 00'	90° 00'
(100):(110)	(001):(021)	53 50	53 33½
(100):(001)	(001):(401)	69 20	70 25½
(100):(101)	(001):(201)	33 30	36 45

*Optics.* Jarlite is biaxial positive or negative with  $V=b[010]$ . A re-determination in sodium light of the indices of refraction by immersion gave values in close agreement with those of Bøgvad:

	$\alpha$	$\beta$	$\gamma$
Bøgvad	1.427	1.432–1.433	1.435
Ferguson	1.430	1.435	1.437±0.002

For meta-jarlite, Bøgvad obtained an average value of  $1.432 \pm 0.004$ . A complete new determination, also by immersion, gave  $\alpha=1.429$ ,  $\beta=1.431$ ,  $\gamma=1.434$ , all  $\pm 0.002$ . Since, as noted above, x-ray powder pictures suggest that meta-jarlite is only a variety of jarlite, the following average refractive indices appear the best for these two minerals when considered as one, jarlite:

$$\alpha=1.429, \beta=1.433, \gamma=1.436, \text{ all } \pm 0.002$$

Using a universal stage and sodium light, Bøgvad measured  $2V$  about  $X$  for six jarlite crystals, and obtained three angles from  $78^\circ 10'$  to  $80^\circ 00'$  (sign negative), and three others from  $91^\circ 40'$  to  $92^\circ 30'$  (sign positive). Three new measurements, also made on the universal stage, gave  $81.1^\circ$  (negative),  $91.95^\circ$  (positive), and  $98.65^\circ$  (positive). Since this angle varies so much, presumably with minor changes in chemical composition, a fair statement of the optic axial angle of jarlite is suggested as

$$2V=90^\circ \pm 10^\circ, \text{ positive or negative}$$

No measurement of this angle has been made for meta-jarlite but, from the straightness of the brushes in the interference figure, it must be approximately  $90^\circ$  as indicated by Bøgvad. This observation is in keeping with the close similarity of meta-jarlite to jarlite in other respects.

Bøgvad established the following incomplete optic orientation of jarlite:  $Y=b[010]$ ; optic axial plane  $\perp c(001)$  (structural setting); and  $X:\perp c(001)=16^\circ$  to  $24^\circ 15'$ . During the present study, these observations were checked and the complete optic orientation was established. For the angle  $X:\perp c(001)$  Bøgvad obtained on three separate crystals the values  $16^\circ$ ,  $16^\circ$ ,  $24^\circ 15'$ . A re-measurement of this angle, also on three separate crystals each mounted for observation along  $[010]$  on the microscope stage, led to the values  $16^\circ$ ,  $17^\circ$ ,  $19\frac{1}{2}^\circ$  all  $\pm 1^\circ$ . In order to determine the angle made by  $X$  with  $c[001]$ , it was necessary to use some method involving a single-crystal x-ray photograph since all crystals showed only



the two basal faces. An attempt was made to mount a crystal for rotation about  $c[001]$  using the known angles  $\beta$  and  $X:\perp c(001)$  in conjunction with the goniometric reflection from a basal face, and assuming one of two possible orientations for  $X:c[001]$ , namely that  $X$  lies either in front of or behind  $c$ . With the latter orientation assumed, the first crystal mounted in this way gave a perfectly centered rotation photograph for a row whose period is  $7.26\text{ kX}$  ( $c[001]=7.24\text{ kX}$ ), thereby proving that this axis is  $c[001]$  and that  $X$  lies *behind*  $c[001]$ . Thus, for an angle  $\beta\ 101^\circ 49'$  (say  $102^\circ$ ) and for an angle  $X:\perp c(001)$  of  $17^\circ$ , the orientation is  $X:c[001] = -(17^\circ - (102^\circ - 90^\circ)) = -5^\circ$ . Or, considering the variation in  $X:\perp c(001)$  as  $16^\circ - 20^\circ$ , this orientation becomes  $X:c[001] = -(4^\circ - 8^\circ)$ .

*Composition, Cell Content, and Specific Gravity.* It was noted above that chemical analyses of jarlite and meta-jarlite by Blix (in Bøggvad, 1933) led to the same ideal formula  $\text{NaSr}_3\text{Al}_3\text{F}_{16}$  for both minerals. With Bøggvad's measured specific gravity of 3.93 and the author's cell dimensions, this formula gives a non-integral number of empirical formula weights in the unit cell ( $Z \sim 4.4$ ). For  $Z=4$ , Bøggvad's ideal chemical formula gives a calculated density of 3.61. Of the various possible sources of error, the specific gravity seemed the most likely, and for this reason was carefully re-determined on three separate jarlite crystals using, as Bøggvad did, Clerici's solution and a Westphal balance. The three new values checked closely: 3.86<sub>2</sub>, 3.86<sub>4</sub>, 3.86<sub>9</sub> giving a new best value of  $3.87 \pm 0.01$ .

With the specific gravity now confirmed, and the correctness of the cell dimensions assured by the indexing of the powder photograph, the only possible conclusion is that the chemical formula,  $\text{NaSr}_3\text{Al}_3\text{F}_{16}$ , is in error. Once again, after this conclusion had been reached, it was found that Brosset (1942) had similarly reached the same conclusion some years earlier.

For meta-jarlite, Bøggvad measured the specific gravity as 3.78, using Clerici's solution. In the present study, relatively large fragments of meta-jarlite were available, and the specific gravity of two of these was measured on the Berman balance. These gave the values 3.59 (on 14 mg.) and 3.65 (on 24 mg.) indicating a minimum value, from the new measurements, of 3.65. The low value is attributed to the abundant inclusions which characterize specimens of meta-jarlite.

#### PROSOPITE— $\text{CaAl}_2(\text{F},\text{OH})_8$

This mineral has been reported from Ivigtut by Bøggvad (1938), who gives no descriptive details. Attempts to find prosopite again in the material available were unsuccessful; however, specimens from two of the well-established localities, Altenberg, Saxony, and St. Peter's Dome, El

Paso County, Colorado, were generously loaned by Professor Clifford Frondel of the Harvard Mineralogical Museum, and these made it possible to study this mineral.

The specimen from Saxony (Harvard 97155) showed relatively large (1 cm.) brownish-gray tabular crystals which proved, from an  $x$ -ray powder photograph, to consist of kaolinite, thus confirming the statements in the standard texts that prosopite from this locality is often largely altered to this mineral. The specimen from Colorado showed small (1 mm.), colorless, transparent, tabular crystals forming a druse on massive pachnolite. Both the morphological and the  $x$ -ray crystallography of these crystals has been studied by the staff at the Harvard labo-

TABLE 4. PROSOPITE— $\text{CaAl}_2(\text{F}, \text{OH})_8$ :  $X$ -RAY POWDER PATTERN

Monoclinic,  $C2/c$ ;  $a=6.67$ ,  $b=11.17$ ,  $c=7.37$  kX,  $\beta=94^\circ 56'$ ;  $Z=4$

$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$
$\frac{1}{2}$	$7.7^\circ$	5.74	(110)	5.711	4	$20.3^\circ$	2.22	(042)	2.223
10	10.2	4.34	(111)	4.355	$\frac{1}{2}$	20.7	2.17	(310)	2.173
2	12.15	3.65	(002)	3.671	6	21.15	2.13	(240)	2.138
1	13.35	3.33	(200)	3.323				(311)	2.133
4	13.75	3.23	(130)	3.248	$\frac{1}{2}$	21.65	2.08	(241)	2.084
5	14.55	3.06	(022)	3.068	3	22.35	2.02	(241)	2.023
$\frac{1}{2}$	14.75	3.02	( $\bar{1}$ 31)	3.018				(151)	2.020
3	15.65	2.85	(220)	2.856	1	23.25	1.947	(312)	1.943
$\frac{1}{2}$	16.3	2.74	( $\bar{2}$ 21)	2.731	5	23.75	1.909	(133)	1.916
$\frac{1}{2}$	17.25	2.59	( $\bar{2}$ 02)	2.576				(330)	1.904
2	18.9	2.37	(202)	2.364	6	24.8	1.833	(004)	1.836
4	19.45	2.31	( $\bar{1}$ 13)	2.312	6	25.15	1.809	(331)	1.811

$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$
$\frac{1}{2}$	$26.15^\circ$	1.744	1	$37.15^\circ$	1.273	$\frac{1}{2}$	$49.75^\circ$	1.007
$\frac{1}{2}$	26.75	1.708	$\frac{1}{2}$	37.65	1.259	1	53.9	0.951
1	27.5	1.665	1	38.4	1.238	1	54.6	0.943
1	27.9	1.643	$\frac{1}{2}$	39.55	1.207	$\frac{1}{2}$	56.55	0.921
1	28.3	1.621	1	40.05	1.195	2	59.5	0.892
2	28.8	1.596	2	40.65	1.180	1	62.5	0.867
3	29.35	1.568	1	42.0	1.149	$\frac{1}{2}$	65.9	0.842
2	30.45	1.517	1	43.2	1.123	$\frac{1}{2}$	67.35	0.833
1	32.7	1.423	$\frac{1}{2}$	43.85	1.110	$\frac{1}{2}$	68.55	0.826
1	33.5	1.393	1	44.75	1.092	1	71.55	0.810
1	34.05	1.373	1	45.6	1.076	1	74.1	0.799
2	34.65	1.352	$\frac{1}{2}$	46.25	1.064	1	74.65	0.797
1	35.5	1.324	$\frac{1}{2}$	47.65	1.040	1	75.15	0.795
2	36.8	1.283	$\frac{1}{2}$	48.8	1.022			

ratory in connection with the preparation of Volume II of the Seventh Edition of Dana's *System of Mineralogy*. The present investigation was confined to verifying the cell dimensions and space-group previously determined at Harvard, and to establishing the indexed powder data.

Rotation and Weissenberg photographs were taken of two of the monoclinic crystals about axes [010] and [110]. These gave the monoclinic cell dimensions:

$$a=6.67, b=11.17, c=7.37, \text{ all } \pm 0.02, \text{ kX}, \beta=94^{\circ} 56' \pm 20'$$

which compare closely with the unpublished values derived at Harvard by Berman & Wolfe (private communication, 1941, kindly made available by Dr. Frondel):

$$a=6.69, b=11.11, c=7.32, \text{ all } \pm 0.03 \text{ kX}, \beta=95^{\circ} 00' \pm 15'$$

The space-group was confirmed as  $C2/c$ . The powder data indexed to  $\theta(\text{Cu}) = 25.15^{\circ}$  on the author's cell dimensions, appear in Table 4. Certain small differences between these values and those given for prosopite from Saxony in the ASTM Card Index (1945, card 539), are probably due to small chemical difference in the material from two localities.



Ralstonite occurs at Ivigtut with thomsenolite and cryolite as small, clear, colorless octahedral and cubo-octahedral crystals. For some time the chemical formula had been in doubt but Pabst (1939), in considering the structure of ralstonite, concluded that the best formula for the material from Ivigtut is  $\text{Na}_{2.8}(\text{Al}, \text{Mg})_{16}(\text{F}, \text{OH})_{48} \cdot 7\text{H}_2\text{O}$ . Since Pabst's work shows the structure to be defective with the 2.8 Na occupying a 16-fold position, and the  $7\text{H}_2\text{O}$  occupying an 8-fold position in the space-group  $Fd3m$ , the formula could be written in the following way to indicate the defective nature of the structure:  $\text{Na}_{16-x}(\text{Al}, \text{Mg})_{16}(\text{F}, \text{OH})_{48} \cdot 8-y\text{H}_2\text{O}$  with  $x \sim 13$  and  $y \sim 1$ . In its simplest terms, this formula reduces to that given at the head of this section with  $x \sim 1.6$  and  $y \sim 0.1$ .

Although earlier goniometric work and Pabst's later x-ray investigation indicated no departure from cubic symmetry, ralstonite has long been known to show "anomalous anisotropism." During the present study, one x-ray powder photograph of a good octahedral crystal gave a pattern which agreed well with Pabst's powder data, except that the new cell-edge, 9.92 kX, is noticeably larger than Pabst's, 9.87 Å. A second powder photograph of another ralstonite crystal, a cubo-octahedron, was similar to the first but showed one notable difference, namely that prac-

TABLE 5. RALSTONITE: COMPARISON OF OBSERVED X-RAY POWDER DATA

$(hkl)$	Pabst (Mo/Zr)		Ferguson (Cu/Ni)			
	(cubic)		(cubic)		(non-cubic)	
	$I$	$d$	$I$	$d$	$I$	$d$
(111)	10	5.68	10	5.70	10	5.77
(113)	8	2.97	9	2.99	9	2.99
(222)	7	2.84	8	2.86	8	2.86
(004)	1	—	1	2.47	1	2.47
(133)	2	2.26	3	2.27	3	2.27
(224)	4	2.01	4	2.03	4	2.02
(115)	6	1.879	7	1.909	7	1.905
(333)						
(044)	7	1.740	8	1.750	8	1.750
(135)	1	1.664	2	1.679	2	1.670
(026)	1	1.553	2	1.568	2	1.561
(335)	4	1.485 $b$	2	1.512	1	1.510
(226)						
(444)	1	1.421	1	1.435	$\frac{1}{2}$	1.429
(117)	3	1.379	4	1.391	3	1.384 $b$
(155)						
(137)	3	1.282	3	1.292	3	1.289
(355)						
(008)	1	1.228	1	1.240	1	1.239
					$\frac{1}{2}$	1.232
(228)	2	1.158	3	1.171	3	1.167
(066)					1	1.159
(266)	1	1.127	2	1.140	1	1.137
					$\frac{1}{2}$	1.129
(048)	1	1.102	1	1.111	1	1.109
(119)					$\frac{1}{2}$	1.102
(357)			$\frac{1}{2}$	1.090	$\frac{1}{2}$	1.084 $b$
(466)			$\frac{1}{2}$	1.058	$\frac{1}{2}$	1.056 $b$
(139)			1	1.042	$\frac{1}{2}$	1.039
					$\frac{1}{2}$	1.033
(448)			$\frac{1}{2}$	1.013	$\frac{1}{2}$	1.011 $b$
			$\frac{1}{2}$	0.998	$\frac{1}{2}$	0.994
			$\frac{1}{2}$	0.974	$\frac{1}{2}$	0.972
			2	0.958	3	0.954 $b$
			1	0.867	$\frac{1}{2}$	0.867
			$\frac{1}{2}$	0.850	$\frac{1}{2}$	0.861
			2	0.842	1	0.851
			2	0.840	1	0.841
			2	0.827	1	0.834 $b$
			1	0.818	$\frac{1}{2}$	0.827
			$\frac{1}{2}$	0.805	$\frac{1}{2}$	0.819 $b$
					$\frac{1}{2}$	0.812

 $b$  = broad band.



tically all lines beyond about  $d=1.30$  kX appeared as doublets or as broadened bands. The extent of the broadening is shown in Table 5, which compares the observed data for the two new ralstonite photographs with those of Pabst.

In view of the optical anisotropism of the mineral, the most likely explanation for such broadening is a slight departure, in this crystal, from cubic symmetry. With this in mind, the author attempted to derive tetragonal (pseudo-cubic) cell dimensions from the powder photograph, but this proved unsuccessful. No further work was done on this crystal, and the only conclusion to be drawn from the line broadening is a suggestion of slight departure from cubic symmetry in that particular crystal of ralstonite.

X-ray powder photographs of four specimens of "hagemannite," a dull, yellow to brown ochre occurring with the cryolite and associated minerals at Ivigtut, confirmed the observation of Frondel (1948) that this "mineral" consists mainly of a variable mixture of ralstonite, thomsenolite, and goethite. The author's photographs indicated a predominance of ralstonite over thomsenolite in all four specimens.

#### WEBERITE— $\text{Na}_2\text{MgAlF}_7$

In 1938 Bøgvad described a new mineral from Ivigtut, weberite, which occurs as mottled-gray translucent masses with the cryolite, chiolite, topaz, etc. Bøgvad (1938) noted no crystals, but he did observe two cleavages, one prismatic and one pinakoidal, both poor. He also determined the specific gravity (2.96) and all the optical constants except the orientation. Brosset (1942) derived the orthorhombic cell dimensions and space-group, and Byström (1944) carried out a complete structure determination.

During the present investigation, the cell dimensions and space-group were re-determined, and the indices of the cleavage and optic orientation were established. Unsuccessful attempts were made to orient fragments for single-crystal photographs, using reflections from the uneven cleavage surfaces. Following this, an effort was made to orient an irregular fragment about one optical direction by adjusting the fragment, on the goniometer head, to extinction in two mutually perpendicular planes, on the stage of a petrographic microscope. For rotation about the  $Z$  optical direction, this gave good rotation and Weissenberg photographs, the rotation axis proving to be  $c[001]$  of Brosset. A second fragment was oriented in a similar manner for rotation about the  $X$  optical direction, the axis in this case proving to be  $a[100]$  of Brosset. The second fragment was not as well centered as the first, and the cell dimensions from it are considered less accurate. Following is a comparison of the cell dimen-

TABLE 6. WEBERITE— $\text{Na}_2\text{MgAlF}_7$ : X-RAY POWDER PATTERN  
Orthorhombic,  $I2mm$ ;  $a=7.29$ ,  $b=7.05$ ,  $c=9.97$  kX;  $Z=4$

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	( <i>hkl</i> )	<i>d</i> (calc.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	( <i>hkl</i> )	<i>d</i> (calc.)
5	7.5°	5.89	(101)	5.885	3	23.3°	1.943	(231)	1.937
6	8.75	5.05	(110)	5.068	1	24.9	1.826	(400)	1.823
3	12.5	3.55	(020)	3.525	10	25.6	1.779	(224)	1.777
$\frac{1}{2}$	14.65	3.04	(103)	3.024	1	26.9	1.699	(233)	1.698
9	15.1	2.95	(202)	2.942	1	27.5	1.665	{(134)}	1.665
9	15.45	2.89	(022)	2.878				{(006)}	1.662
$\frac{1}{2}$	17.6	2.54	(220)	2.534	5	29.9	1.542	{(305)}	1.542
$\frac{1}{4}$	18.9	2.37	(301)	2.361				{(422)}	1.540
5	19.5	2.30	(213)	2.319	4	30.5	1.515	{(206)}	1.512
4	20.05	2.24	{(114)}	2.237				{(242)}	1.512
			{(130)}	2.237	$\frac{1}{2}$	31.4	1.475	(404)	1.471
$\frac{1}{2}$	22.0	2.05	{(204)}	2.058	1	32.2	1.443	{(501)}	1.443
			{(024)}	2.035				{(044)}	1.439
			{(303)}	1.962					
3	23.0	1.967	{(321)}	1.962					

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)
$\frac{1}{2}$	33.45°	1.395	$\frac{1}{2}$	42.9°	1.129	$\frac{1}{2}$	60.9°	0.880
1	35.05	1.339	1	43.35	1.120	$\frac{1}{2}$	62.0	0.871
1	37.2	1.271	$\frac{1}{2}$	44.6	1.095	2	63.75	0.857
$\frac{1}{2}$	38.0	1.249	$\frac{1}{2}$	47.35	1.045	3	64.6	0.851
$\frac{1}{2}$	38.6	1.232	$\frac{1}{2}$	48.3	1.030	3	65.3	0.846
$\frac{1}{2}$	39.35	1.212	1	48.85	1.021	2	67.3	0.833
$\frac{1}{2}$	40.6	1.181	2	51.55	0.982	1	71.5	0.811
$\frac{1}{2}$	41.35	1.164	1	52.55	0.968	2	73.6	0.801
3	42.0	1.149	$\frac{1}{2}$	59.7	0.890			

sions of Brosset with the best of those derived from the two new sets of photographs:

	<i>a</i>	<i>b</i>	<i>c</i>
Brosset	7.29	7.05	9.97 Å
Ferguson	7.30	7.05	9.97 kX

The new values confirm Brosset's cell dimensions which are accepted for weberite in Table 6, giving the x-ray powder data indexed to  $\theta(\text{Cu}) = 32.2^\circ$ .

Brosset's extinction conditions led to the possible space-groups  $Immm$ ,  $I222$ ,  $I2_12_12_1$ ,  $I2mm$ , and Byström's structure is based on  $I2mm$ . Both investigators apparently observed only one weak exception to the condition  $(0kl)$  present only with  $k=2n$  and  $l=2n$ , noting  $(0kl)$  present

only with  $(k+l) = 2n$  and arriving at the above as possible space-groups. The author could detect no exceptions to the first condition on either set of Weissenberg photographs, and so deduced as possible space-groups *Ibmm* and *Ibm2*.

The optical character of the rotation axes of both fragments used for the x-ray photographs gave the optical orientation which is combined with the optical observations of Bøgvad as follows:

	(Na)	
$X = a[100]$	$\alpha$ 1.346	Positive
$Y = b[010]$	$\beta$ 1.348	$2V = 83^\circ \pm 3^\circ$
$Z = c[001]$	$\gamma$ 1.350	

To determine the indices of the cleavage planes, a zero-layer Weissenberg photograph was taken about a random axis in the cleavage plane of one fragment. The various orders of reflection proved to be from planes (101), and thus the cleavage of weberite may be described as prismatic  $\{101\}$ , poor. The calculated acute cleavage angle (101):(10 $\bar{1}$ ) about  $c[001] = Z$ , is  $72^\circ 21'$  which is in excellent agreement with the acute cleavage angle about  $Z$  measured by Bøgvad in thin section,  $72^\circ$ . The second less conspicuous cleavage noted by Bøgvad as parallel to the optic axial plane would have indices  $\{010\}$ ; this cleavage was not observed by the author.

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# OWYHEEITE<sup>1</sup>

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Owyheeite ( $5\text{PbS} \cdot \text{Ag}_2\text{S} \cdot 3\text{Sb}_2\text{S}_3$ ) is a rare mineral sulpho-salt, descriptions of which are somewhat incomplete. New data obtained by means of x-ray diffraction in the laboratories of the Department of Mineralogy, Queen's University, are presented below; these not only confirm the validity of owyheeite as a mineral species but also indicate that another rare sulpho-salt, warrenite, is identical with it.

Owyheeite was first described by Burton (1868) as argentiferous jamesonite. Subsequently material from the same district (Owyhee Co., Idaho) was analysed and described by Shannon (1920, 1921), who gave it the name owyheeite. Later references to owyheeite add little to the description of the mineral, except those of Ramdohr (1937) and Short (1940) who describe etch-reactions and other data obtained from polished sections.

Eakins (1888) described a new sulpho-salt with a formula  $3(\text{Pb},\text{Fe})\text{S} \cdot 2\text{Sb}_2\text{S}_3$  which he subsequently named warrenite (Eakins 1890). On the basis of similarity in analysis and cleavage, Spencer (1907) identified warrenite as jamesonite. X-ray powder photographs of specimens of warrenite from the type locality (Gunnison Co., Colorado) show that most of them are owyheeite, three are jamesonite, and one is boulangerite.

*Material and Acknowledgments.* The writer first became interested in the mineral when specimens collected by Mr. S. M. Roscoe from the Slocan Rambler Mine, British Columbia, and reported to be owyheeite, were turned over to him for examination. Subsequently, the following specimens were borrowed for study; the original name as given on the label is retained for each specimen.

1. Owyheeite (U.S.N.M., 94054), Poorman Mine, Silver City district, Owyhee Co., Idaho; massive material in a quartz gangue with some pyrite. This is type material described by Shannon (1920).

2. Warrenite (U.S.N.M., 48412), Domingo Mine, Gunnison Co., Colorado; blue to yellow-gray hairlike crystals coating fractures in country rock. This is original type material described by Eakins (1888).

3. Warrenite (U.S.N.M., R998), Domingo Mine, Gunnison Co., Colorado (Roebeling collection); fine hairlike crystals in country rock.

4. Warrenite (U.S.N.M., 82621), Garfield (King Cole) Mine, Cascade Mts., Gunnison Co., Colorado; hairlike to acicular crystals in vugs in country rock, collected by Cross and Smith.

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5. Warrenite (U.S.N.M., 86844), Colorado; fine hairlike needles in country rock (type material).
6. Owyheeite (R.O.M., M13021), Poorman Mine, Owyhee Co., Idaho; massive material in quartz.
7. Warrenite (R.O.M., M4063), Domingo Mine, Gunnison Co., Colorado; fine hairlike crystals coating country rock.
8. Warrenite (H.M.M., 82669), Domingo Mine, Gunnison Co., Colorado; fine hairlike needles on country rock.
9. Warrenite (H.M.M., 87798), Domingo Mine, Gunnison Co., Colorado; fine hairlike needles in crevices in country rock.
10. Warrenite (B.M., 84697), Domingo Mine, Gunnison Co., Colorado; a few fine hair-like crystals in a glass vial. This is the material described by Spencer (1907).

For the specimens from the United States National Museum, the writer is indebted to Dr. W. F. Foshag. Specimens from the Royal Ontario Museum were provided through the kindness of Dr. V. B. Meen. Material from the collections of the Harvard Mineralogical Museum was loaned through the courtesy of Dr. C. Frondel, and Dr. F. A. Bannister was kind enough to provide the specimen from the British Museum. The writer also wishes to express his appreciation to Dr. L. G. Berry of Queen's University for assistance, advice, and criticism.

*Physical Properties.* Owyheeite occurs in quartz gangue in massive to coarsely fibrous habit and on country rock as masses of felted hairlike crystals coating crevices and fractures. Some single crystals were found embedded in quartz but none of the material examined proved to be suitable for measurement of interfacial angles. Needles are striated longitudinally and are very brittle due to basal cleavage. The mineral is light silvery gray, frequently tarnished to a blue or yellow tint.

In polished section it is slightly pleochroic and markedly anisotropic from yellowish white to gray. Hardness is slightly less than that of galena. The streak is a bright reddish brown. The writer was unable to obtain a satisfactory microchemical test for silver from the Slocan material but a semi-quantitative analysis indicated a silver content of between five and ten per cent. Standard etch-tests are not determinative; their results agree closely with those listed by Short (1940) except that a reaction with KOH was obtained yielding a vari-coloured coating after two to three minutes.

The specific gravity of owyheeite as measured by Burton (1868) is 6.03; this value is quoted in Palache, Berman & Frondel (1944). The Slocan material on which Dr. Berry was kind enough to make new determinations of specific gravity, was found to contain finely disseminated impurities, principally quartz, pyrite and a black non-metallic mineral. Several determinations were made, the lowest of which were below 6.0. However the three highest values were obtained from the cleanest ma-

terial; on 4.73 mg. the specific gravity was 6.02, this material was then repicked and a cleaner residue weighing 1.58 mg. yielded specific gravities of 6.22 and 6.51. The average of these three values is  $6.25 \pm 0.2$ ; since the impurities are minerals of lower specific gravity it is probable that the true value of owyheeite is slightly higher than this average.

*Structural Crystallography.* There is no recorded occurrence of crystals of owyheeite on which interfacial angles can be measured. Accordingly single crystal rotation photographs and Weissenberg resolutions were made about the needle-axis  $c[001]$  only. Rotation photographs were made from a fragment of the Slocan material, from a hairlike crystal of warrenite (U.S.N.M., 82621) and from a similar crystal of warrenite (B.M., 84697). Zero layer Weissenberg resolutions were made from the first two of these specimens.

Rotation photographs about  $c[001]$  show strong layer lines and very weak intermediate layer lines yielding a pseudo-period  $c' = c/2 = 4.095 \text{ \AA}$ . Since it was found to be impracticable to resolve the true first layer line, a Weissenberg resolution of the first layer line of the pseudo-cell was made from the Slocan fragment. These photographs indicate that owyheeite is orthorhombic and yield the following dimensions<sup>3</sup> of the unit cell:

$$a = 22.82 \quad b = 27.20 \quad c = 8.19 \text{ \AA}$$

Systematically missing spectra for the pseudo-cell  $abc'$  conform to those for the space group  $Pnam - D_{2h}^{16}$ . The axial ratio becomes:

$$a:b:c = 0.8390:1:0.3011$$

X-ray powder photographs of the three specimens of owyheeite are identical with those of warrenite specimens U.S.N.M., R998, 82621, and R.O.M., M4063. There was insufficient material for a powder photograph to be made of warrenite from the British Museum; its identity with owyheeite was established on the basis of rotation photographs. Material from warrenite specimens U.S.N.M., 48412 and H.M.M., 82669 and 87798 yielded x-ray photographs identical with those of jamesonite. Material from warrenite specimen U.S.N.M., 86844 gives a powder photograph identical with that of boulangerite.

Table 1 gives the x-ray powder pattern of owyheeite indexed to  $\theta(\text{Cu}) = 15.75^\circ$  with reference to the pseudo-cell  $abc'$ .

Table 2 presents the two available analyses of owyheeite and the ideal composition for  $\text{Pb}_5\text{Ag}_2\text{Sb}_6\text{S}_{15}$  together with the corresponding numbers of atoms in the pseudo-cell  $abc'$ . Iron and copper have been included with silver and a specific gravity of 6.25 was used in the calculations. The

<sup>3</sup> Using  $\lambda \text{ CuK}\alpha = 1.5418 \text{ \AA}$  and the mass factor 1.6602.

TABLE 1. Owyheelite:  $5\text{PbS} \cdot \text{Ag}_2\text{S} \cdot 3\text{Sb}_2\text{S}_3$ . X-RAY POWDER PATTERN  
Orthorhombic,  $Pnam$ ;  $a=22.82$ ,  $b=27.20$ ,  $c/2=4.095$  Å;  $Z=4$

$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$
$\frac{1}{2}$	10.6°	4.19 Å	(260)	4.215 Å				(051)	3.271 Å
$\frac{1}{2}$	11.0	4.04	(011)	4.050				(280)	3.258
2	11.35	3.92	(450)	3.942	10	13.7°	3.25 Å	(710)	3.241
			(360)	3.898				(151)	3.240
			(170)	3.847				(421)	3.233
1	11.65	3.82	(211)	3.817				(251)	3.145
			(600)	3.803	1	14.2	3.14	(431)	3.123
			(540)	3.797				(650)	3.122
			(221)	3.709				(161)	3.011
2	12.05	3.69	(131)	3.682	2	14.85	3.01	(351)	3.008
			(270)	3.679				(190)	3.004
			(620)	3.671				(441)	2.991
1	12.45	3.57	(311)	3.575				(290)	2.921
			(460)	3.552	5	15.4	2.90	(660)	2.917
			(630)	3.515				(531)	2.892
7	12.75	3.49	(550)	3.501				(800)	2.852
			(321)	3.488	6	15.75	2.84	(810)	2.842
			(180)	3.373				(451)	2.839
4	13.2	3.37	(241)	3.352					
			(331)	3.351					

$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$	$I$	$\theta(\text{Cu})$	$d(\text{meas.})$
$\frac{1}{2}$	16.25°	2.75 Å	5	20.2°	2.23 Å	3	26.42°	1.732 Å
$\frac{1}{2}$	16.65	2.69	3	21.25	2.13	1	27.3	1.681
1	17.15	2.61	1	21.5	2.10	$\frac{1}{2}$	28.3	1.626
$\frac{1}{2}$	17.6	2.55	6	22.05	2.05	1	30.85	1.503
$\frac{1}{2}$	18.35	2.45	2	22.5	2.01	1	31.7	1.467
$\frac{1}{2}$	18.8	2.39	1	23.35	1.945	$\frac{1}{2}$	32.1	1.451
1	19.35	2.33	2	24.42	1.864	1	34.52	1.360
2	19.75	2.28	3	25.85	1.768			

TABLE 2. OWYHEEITE: ANALYSES AND CELL CONTENT

1			2		A	B	
Pb	40.77	18.67	43.86	20.25	19.46	20	42.18
Ag	7.40	6.51	6.14	5.45	8.15	8	8.80
Cu	0.75	1.13	1.55	2.33			
Fe	0.46	0.79	0.05	0.09			
Sb	30.61	23.85	29.26	23.01	23.43	24	29.41
S	20.81	61.62	19.06	57.00	59.31	60	19.61
100.80			99.92			100.00	

1. Poorman mine, Owyhee Co., Id., Shannon (1920). 2. Sheba mine, Star City, Nev., Burton (1868). A. Average content of unit cell. B. Cell content and percentage composition for  $4(5\text{PbS} \cdot \text{Ag}_2\text{S} \cdot 3\text{Sb}_2\text{S}_3)$ .

average content of the pseudo-cell  $abc'$  is nearly  $4(5\text{PbS} \cdot \text{Ag}_2\text{S} \cdot 3\text{Sb}_2\text{S}_3)$  which gives a calculated specific gravity of 6.43. The true cell  $abc$  contains  $8(5\text{PbS} \cdot \text{Ag}_2\text{S} \cdot 3\text{Sb}_2\text{S}_3)$ .

Although the name warrenite has precedence in time, having been proposed by Eakins in 1890, the material described by him was in fact jamesonite. Subsequently the name warrenite was applied erroneously to owyheeite. It seems proper, therefore, to retain the name owyheeite, which was proposed for the silver-bearing mineral, and to discard the name warrenite.

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# HEMATITE-GOETHITE RELATIONS IN NEUTRAL AND ALKALINE SOLUTIONS UNDER PRESSURE

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## ABSTRACT

A series of experiments on the stability of goethite ( $\alpha\text{-FeO}\cdot\text{OH}$ ) shows that it decomposes to hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) in neutral solutions above  $125 \pm 15^\circ\text{C}$ . and in alkaline solutions above  $165^\circ \pm 5^\circ\text{C}$ . at a pressure near the vapour pressure of the solutions. Increase of pressure raises the decomposition temperature of goethite less than  $5^\circ\text{C}$ . per 1000 atmospheres, up to 2000 atmospheres. The possible use of the pressure insensitive pair, hematite-goethite, in geothermometry is discussed.

## INTRODUCTION

A considerable amount of experimental work has been done on the stability relations of the ferric oxides at various temperatures, with especial reference to the occurrence of hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ), the less stable ferro-magnetic maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ), and the polymorphic monohydrates, goethite ( $\alpha\text{-Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$  or  $\alpha\text{-FeO}\cdot\text{OH}$ ) and lepidocrocite ( $\gamma\text{-Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$  or  $\gamma\text{-FeO}\cdot\text{OH}$ ). Posnjak and Merwin (1919) showed in the first systematic study of ferric oxide and its hydrates that the monohydrate was the only one that can be considered to be a compound. Other hydrous varieties are turgite, which was shown to be hematite plus adsorbed water, and limonite, to be goethite with adsorbed water. Maghemite occurs as an oxidation product of magnetite, or as a product of dehydration of lepidocrocite. Maghemite is transformed at high temperatures into the much more stable hematite. No demonstration of the transformation of lepidocrocite into goethite appears to have been published. The synthesis of another monohydrate, *in vitro*, was described by Weiser and Milligan (1935) who called it  $\beta\text{-Fe}_2\text{O}_3\cdot\text{H}_2\text{O}$  (or  $\beta\text{-FeO}\cdot\text{OH}$ ). On boiling it is dehydrated into hematite. Thus the ultimate product of heating any one of the three polymorphic monohydrates is hematite. In the present study only hematite and goethite will be considered.

Posnjak and Merwin (1922) succeeded in synthesizing crystalline goethite in the system,  $\text{Fe}_2\text{O}_3\text{-SO}_3\text{-H}_2\text{O}$ . It resembled microscopically natural fibrous goethite and appeared "only below the temperature of  $130^\circ\text{C}$ . in individual or clustered yellow spherical grains less than 0.01 mm. in diameter." All of the preparations from which the monohydrate was obtained were in the furnace "for several weeks and had become compact and granular. The precipitate which formed when the tubes were first filled were flocculated and showed no evidence of crystallinity.

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TABLE 1. SUMMARY OF PREVIOUS WORK

Reference	Container	Reaction	Starting Materials	Nature of Medium
Posnjak & Merwin (1919)	Open weighing glasses	Decomposition	Natural bladed goethite	Dried air
			Natural fibrous goethite	Dried air
			Microcrystalline "limonite"	Dried air
Posnjak & Merwin (1922)	Sealed Jena tubes $\frac{1}{2}$ full	Decomposition	Ferric sulphate	Very dilute solutions
Tunell & Posnjak (1931)	—	Decomposition	Natural goethite	N/10 HCL solution
Böhm, J. (1925)	—	Boiling	Amorphous hydrated ferric oxide (brown gel)	H <sub>2</sub> O or solutions of KCl or NH <sub>4</sub> Cl
	Autoclave	Aging	Brown gel	2N KOH
Katsurai & Watanabe (1930)	Autoclave	Decomposition	Gelatinous Fe (OH) <sub>3</sub>	Very dilute HCl solution
	Autoclave	Hydrolysis	FeCl <sub>3</sub> solution	0.5 N 0.25 N 0.2 N 0.1 N
Hüttig & Garside (1929)	—	Decomposition	Natural Lepidocrocite	—
			Natural Goethite	—
Gruner (1931)	Sealed glass tubes	Decomposition	Natural Goethite	Distilled water
Williams & Thewlis (1931)	—	Decomposition	Artificial Lepidocrocite	—
Nicholas, Kraemer & Bailey (1932)	—	Hydrolysis	Ferric chloride	0.005 M 0.037 M
Weiser & Milligan (1935)	—	Aging	Fresh amorphous brown gel	Distilled water
		Rapid hydrolysis	Ferric salts	Water solution
		Slow hydrolysis	Ferric chloride	Water solution
		Slow hydrolysis	Other Ferric salts	Water solution
		Decomposition	FeO · OH	Dried air

pH of Medium	Temp. ° C.	Pressure	Length of Experiment	Products	Identification
—	210–220	—	A number of weeks	Hematite	X-ray
—	150–215	—	A number of weeks	Hematite	X-ray
—	145–155	—	A number of weeks	Hematite	X-ray
acid	up to 130	Vapor pressures	8 to 10 weeks	Goethite	X-ray
acid	About 100	—	"A few weeks"	Hematite	X-ray
acid	About 100	—	—	Hematite	X-ray
alkaline	150	—	Nearly 2 hours	Goethite	X-ray
acid	150	—	1 hour	Hematite	X-ray
acid	120	—	1 hour(?)	Hematite	X-ray
acid	140 & 150	—	1 hour	Hematite	X-ray
acid	160	—	1 hour(?)	Hematite	X-ray
acid	180–185	—	1 hour(?)	Hematite	X-ray
—	240	10 mm.	—	Maghemite	Colour
—	275	10 mm.	—	Hematite	Colour
Neutral	200	Vapour pressures	Over 90 days	Hematite	X-ray
	250				
	275				
—	250–300	—	—	Maghemite	—
	500–600	—	—	Hematite	—
acid	100	—	1 hour	Amorphous	X-ray
			24 hours	Hematite	X-ray
acid	100	—	1 hour	Hydrous Fe <sub>2</sub> O <sub>3</sub> of unknown identity	X-ray
			8 hours	Hematite	
—	25	—	few weeks	Hematite	X-ray
	near b.p.	—	few hours	Hematite	X-ray
acid	25	—	—	Hematite	X-ray
acid	25	—	—	β-FeO · OH	X-ray
acid	25	—	—	Goethite	X-ray
—	150–184	—	—	Hematite	X-ray

As heating progressed nuclei formed in the solution, not only within the limits of the tube occupied by the precipitate, but, especially at higher temperatures, above the original precipitate. The nuclei became centers of spherulites which, especially at higher temperatures, grew to determinable sizes or disappeared while larger ones grew."

At the boiling temperature of about 1/10 normal HCl solution, natural goethite was decomposed into hematite in a few weeks (Tunell and Posnjak, 1931). Tunell and Posnjak held that this conversion at practically 100° C. does not conflict with the higher transition temperature in the binary system,  $\text{Fe}_2\text{O}_3\text{-H}_2\text{O}$ , obtained as a limit in the system,  $\text{Fe}_2\text{O}_3\text{-SO}_3\text{-H}_2\text{O}$ , since the hematite and goethite fields may overlap more in the system,  $\text{Fe}_2\text{O}_3\text{-HCl-H}_2\text{O}$ , than in the system,  $\text{Fe}_2\text{O}_3\text{-SO}_3\text{-H}_2\text{O}$  and further experimental work on the former system may show the transition temperature to be the same in both systems. Böhm (1925) obtained similar results in pure water or solutions of KCl or  $\text{NH}_4\text{Cl}$ .

The reactions in the system,  $\text{Fe}_2\text{O}_3\text{-H}_2\text{O}$  are extremely slow and are not promptly reversible. Ferric oxide under ordinary conditions apparently cannot be hydrated. Most of the time spent by Posnjak and Merwin (1919) in the dehydration experiments was used to decompose the substance undergoing test. "Decomposition proceeds at an exceedingly slow rate at the temperature at which it is first discovered and it was necessary to raise the temperature considerably to dehydrate the substance within a reasonable period." In experiments made in open weighing glasses with no regard for the pressures, they found that complete decomposition of well crystallized and optically amorphous goethite took place in several cases below 200° C. Dehydration data of Posnjak, Merwin, and other workers are summarized in Table 1.

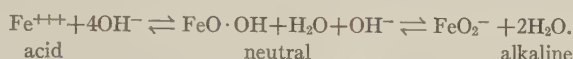
About 50 experiments by Gruner (1931), some of them over periods of 90 days, in pure water showed that goethite is unstable at 200° C. In no case was complete dehydration obtained but 2.5 to 3% of water remained with the product.

The remainder of the experiments summarized in Table 1 were done over periods of the order of an hour and all but one were carried out either in pure water or acid solutions. The exception is that of Böhm (1925) who obtained goethite by heating amorphous hydrated ferric oxide in 2N KOH solution in an autoclave at 150° C. for nearly two hours. Katsurai and Watanabe (1930) also made autoclave runs and used  $\text{FeCl}_3$  solutions of various strengths to obtain hematite (rouge) at as low as 120° C. by hydrolysis of 0.5N  $\text{FeCl}_3$ . They made hematite from gelatinous ferric hydroxide in very dilute HCl solution by heating at 150° C. for one hour.

The qualitative effect of pH on the decomposition of iron oxide monohydrate is shown in Table 1. In acid solutions the decomposition tempera-



ture is near 100° C., and in alkaline solutions is above 150° C. The increase in the decomposition temperature in acid solutions up to approximately 130° C. in neutral solutions is summarized by Tunell and Posnjak (1931). The exact relations, however, are uncertain. In acid and neutral solutions, iron oxides are in equilibrium with the solutions containing ferric iron ions ( $\text{Fe}^{+++}$ ), but in alkaline solutions, they can be considered to be in equilibrium with solutions containing ferrate ions ( $\text{FeO}_2^-$ ), or some derivative of ferrate ions. The equilibrium would then be summarized as follows:



If the iron oxide monohydrate has a hydroxyl group in the unit cell, *i.e.*, its formula is  $\text{FeO} \cdot \text{OH}$ , it should be more stable in alkaline solutions. Bragg (1937) suggested on the basis of Ewing's work that the formula of goethite is  $\text{HFeO}_2$  while that of lepidocrocite is  $\text{FeO} \cdot \text{OH}$ . If this is correct, then lepidocrocite should be more stable in alkaline solutions. However, Peacock recently (1942) gave the hydroxide form to both monohydrates: goethite,  $\alpha\text{-FeO} \cdot \text{OH}$  and lepidocrocite  $\gamma\text{-FeO} \cdot \text{OH}$ . It would then appear that both forms of monohydrate are more stable in alkaline than in acidic solutions, but their relationship with each other is still unknown.

The effect of pressure as an independent variable of the hematite-goethite equilibrium has not yet been systematically studied. In two of the experiments listed in Table 1, by Hüttig and Garside (1929), the pressure was measured, but it was only 10 mm. in each case. In other cases the pressures were either vapor pressures or not reported at all. Gruner (1931) pointed out that the molecular volumes of hematite, water, and goethite are 30.38, 18.00, and 41.58, respectively and he suggested that the decomposition point of goethite would be raised by higher pressures.

It was concluded that further data on the decomposition temperature of goethite would be useful, especially at elevated pressures, and including alkaline as well as neutral water solutions in the system.

### EXPERIMENTS

A summary of the experimental data and results is shown in Table 2. The first six experiments were made in a one-litre Cook autoclave. In all but one of these six runs, the pressure was the vapour pressure of the closed  $\text{Fe}_2\text{O}_3\text{-H}_2\text{O}$  system at the temperature of experiment. In the other autoclave run, nitrogen gas supplied the pressure. Heating was by means of a gas burner and the temperatures were measured with a mercury thermometer. The remaining thirty-one runs were carried out in a pres-

TABLE 2A

Exp. No. (Series Fe)	Bomb Charge					Equi- librium Temper- ature ° C	Calcu- lated Pres- sure Atm.	Time of Heat- ing
	Iron Oxide Solid			Aqueous Liquid Medium				
	Form	Weight grams	H <sub>2</sub> O in Iron Oxide %	Compo- sition	Deg- ree of Fill- ing %			
1	Hematite	10.0	—	H <sub>2</sub> O	80	108 ± 1	1.3	5 hours
2	Goethite	10.0	—	H <sub>2</sub> O	80	141 ± 1	3.6	24 hours
3	Goethite	10.0	—	H <sub>2</sub> O	80	140 ± 1	25.0	10 hours
4	Goethite	10.0	—	H <sub>2</sub> O	80	170 ± 1	7.8	18 hours
5	Goethite	10.0	—	H <sub>2</sub> O	80	200 ± 1	15.3	18 hours
6	Goethite	10.0	—	H <sub>2</sub> O	80	227 ± 3	27.6	24 hours
7	Hematite	6.0	12.20	H <sub>2</sub> O	67	435 ± 1	1500	44 hours
8	Hematite	6.0	12.20	H <sub>2</sub> O	67	300 ± 1	80	40 hours
9	Goethite	6.0	12.60	H <sub>2</sub> O	67	215 ± 10	20	36 hours
10	Goethite	6.0	12.60	H <sub>2</sub> O	83	300 ± 1	1080	3 days
11	Goethite	6.0	12.60	H <sub>2</sub> O	88	255 ± 1	1130	4 days
12	Goethite	6.0	12.60	H <sub>2</sub> O	93	205 ± 1	1250	7 days
13	Goethite	6.0	12.60	H <sub>2</sub> O	91	230 ± 1	1700	7 days
14	Goethite	6.0	12.60	0.1M NaOH	93	227 ± 4	1600	3 days
15	Goethite	6.0	12.60	"	93	210 ± 1	1300	2 days
16	Goethite	6.0	12.60	"	96	270 ± 1	2320	10 hours
17	Goethite	6.0	12.60	"	96	192 ± 1	1400	3 days
18	Goethite	6.0	12.60	"	93	205 ± 1	1200	2½ days
19	Goethite	6.0	12.60	"	93	196 ± 3	1200	2 days
20	Goethite	6.0	12.60	"	91	194 ± 2	600	3 days
21	Hematite from Exp. 13	2.0	1.08	"	91	192 ± 1	600	2 days
22	Goethite	2.0	12.60	"	91	200 ± 2	650	4 days
23	Goethite	2.0	12.60	"	88	195 ± 2	250	5 days
24	Goethite	2.0	12.60	"	91	173 ± 3	250	5 days
25	Goethite	2.0	12.60	"	92	164 ± 2	50	5 days
26	Goethite	2.0	12.60	"	90	171 ± 1	90	7 days
27	Goethite	2.0	12.60	"	98	192 ± 1	1900	6 days
28	Goethite	2.0	12.60	"	99	181 ± 1	2000	6 days
29	Goethite	2.0	12.60	"	100	160 ± 3	1860	5 days
30	Goethite	2.0	12.60	"	100	142 ± 1	1600	7 days
31	Hematite	2.0	11.92	"	100	142 ± 1	1600	6 days
32	Hematite	2.0	11.92	"	99	150 ± 1	1700	7 days
33	Hematite	2.0	11.92	"	95	145 ± 1	500	7 days
34	Goethite	2.0	12.20	"	94	150 ± 1	500	7 days
35	Goethite	2.0	12.20	"	93	159 ± 1	500	6 days
36	Goethite	2.0	12.20	"	93	165 ± 1	500	8 days
37	Hematite	6.0	11.92	"	100	167 ± 1	2000	5 days

TABLE 2B

Exp. No.	Bomb Products			
	Iron Oxide Solid			Remarks
	Form	Method of Deter- mination	H <sub>2</sub> O in Iron Oxide %	
1	Goethite	X-ray	—	Orange coloured fine-grained solid and solu- tion.
2	Hematite	X-ray	—	Bright red solid.
3	Hematite	colour	—	Bright red solid, hard to filter.
4	Hematite	colour	—	
5	Hematite	colour	—	Bright red solution. Very finely divided solid.
6	Hematite	colour	—	
7	Hematite	colour	0.00	Solution was red. Bright red precipitate.
8	Hematite	colour	1.15	
9	Hematite	colour	6.37	
10	Hematite	colour	1.27	Bright red solid.
11	Hematite and Goethite	colour	0.06	Bright red mass with yellow specks of go- ethite.
12	Hematite and Goethite	colour	8.09	Yellow solid with red particles of hematite.
13	Hematite	colour	1.08	Temp. rose from 230° to 263° in last 12 hours.
14	Hematite	colour	3.68	Solid is dark red exactly as in natural hema- tite. Clear solution.
15	Hematite	colour	0.49	Dark red present. Clear solution.
16	Hematite	colour	1.73	
17	Hematite and Goethite	colour	9.80	Yellow product with red specks of hematite.
18	Hematite	tabular crystals	0.41	Mass of red crystalline hematite. Clear solution.
19	Hematite and Goethite	colour	8.36	Yellow solid with very minute red hematite specks.
20	Hematite and Goethite	colour	7.00	Brown mass. Clear solution.
21	Hematite	colour	1.35	Dark red product.
22	Hematite	colour, crystals	2.50	Red crystalline mass. Clear solution.
23	Hematite	colour	0.50	
24	Hematite and Goethite	colour	4.86	Brownish-red product mostly hematite. Few flecks of goethite in bottom.
25	Goethite	colour	11.77	Yellow, same colour as starting solid.
26	Hematite and Goethite	colour	6.12	Pale yellow solution. Top of solids is red but mass is otherwise yellow-brown.
27	Hematite	colour	0.82	Clear solution. Reddish brown mass with yellow lumps.
28	Hematite and Goethite	colour	4.68	Reddish solution. Lumps of original charge coated with red hematite.
29	Hematite and Goethite	colour	—	Red solution. Very fine orange-red residue.
30	Goethite	colour	10.60	Clear solution. Residue all yellow from top to bottom.
31	Hematite and Goethite	colour	8.00	Clear solution. Film of hematite on top of yellow mass. Upper sides of pot and lid show yellow particles.
32	Hematite and Goethite	colour	11.05	Clear solution. Reddish brown mass.
33	Hematite and Goethite	colour	—	Clear solution. Layer of yellow patches on top of brown mass.
34	Goethite	colour	11.50	Solution clear. Product all yellow.
35	Goethite	colour	12.30	Clear solution. Product all yellow.
36	Goethite	colour	11.02	Green solution. Yellowish-brown mass.
37	Hematite	colour, crystals	0.25	Clear solution.

sure bomb which has been described previously (Smith, 1947). The heating was by means of an electrical resistance jacket furnace. The temperatures were measured with a chromel thermocouple and a potentiometer.

All of the experimental runs consisted of adding a weighed amount of prepared iron oxide material to a measured volume of the liquid in the autoclave or bomb, sealing the vessel, bringing the system up to the desired temperature, keeping the system at that temperature for a certain period of time, and finally cooling the sealed vessel back to room temperature.

In the bomb runs, the charge was contained in a graphite pot with lid to minimize the possible formation of magnetite by the action of the solutions on the bomb wall. The inner volume of the graphite pot with the lid on was 82.2 cc. The graphite pot was kept saturated with water between runs.

An indirect method was used to measure the pressure of the system at the maximum temperature of experiment. It was assumed that the liquid phase in the system had the thermal expansion properties of pure water and the pressures for the various degree of filling of the bomb and the experimental temperatures were read directly from a chart showing the relations between specific volume of water, temperature, and pressure, as compiled by Béland (1948).

The runs described in Table 2 can be grouped according to the iron oxide material added to the solution. A brown iron oxide gel was prepared by mixing 10 litres of distilled water, 540 grams of ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , Baker's analysed reagent) and 3 litres of 15N ammonium hydroxide, in that order. A yellow iron oxide gel was made from 225 grams of ferrous chloride (Baker's analysed reagent) in 5 litres of distilled water, to which were added 170 grams of ammonium carbonate in 4 litres distilled water to produce ferrous carbonate, which was then oxidized to the monohydrate by 600 cc. of 3% hydrogen peroxide. After standing overnight, each gel was washed until they gave negative tests with silver nitrate. X-ray powder photographs of them showed the brown gel to be pure hematite and the yellow gel to be pure goethite. A portion of the brown and yellow gels, dried at 105° C., gave red and yellowish brown streaks, respectively.

The runs described in Table 2 A and B can also be grouped according to the composition of the solution in contact with the iron oxide. The earlier experiments were carried out in pure water, the later ones in 0.1M NaOH solution. The recrystallization and conversion of the iron oxide material added to the charge was found to be very much more rapid in the alkaline solutions.

The brown iron oxide gel (hematite x-ray pattern), left to stand in



water at room temperature ( $20^{\circ}$ – $27^{\circ}$  C.) for 2 years, assumed a bright yellow color. It gave the goethite x-ray powder diffraction pattern.

### DISCUSSION OF EXPERIMENTAL RESULTS

Our results are in accord with those of Posnjak and Merwin (1919) and of Böhm (1925), in that there is an increase in the decomposition temperature of goethite with increasing  $pH$ . By projecting back to negligible pressure on the  $t-p$  plot, we believe that in pure water the decomposition temperature of goethite is  $125^{\circ} \pm 15^{\circ}$  C., and in 0.1M NaOH

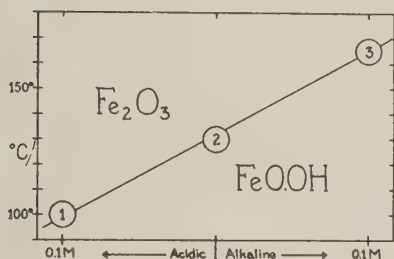


FIG. 1

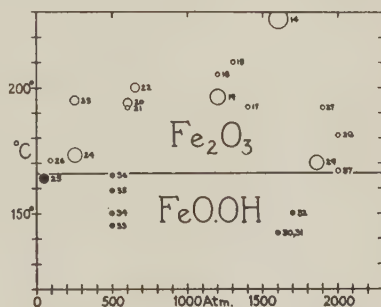


FIG. 2

FIG. 1. The decomposition temperature of goethite in contact with water solution at a pressure of 1–2 atmospheres, as a function of the acidity-alkalinity of the solution. Point 1 is from data of Tunell and Posnjak (1931), Point 2 is from data of Posnjak and Merwin (1922), and Point 3 was located by extrapolating to low pressure from our data.

FIG. 2. The stability field boundary, hematite-goethite, in 0.1 M NaOH solution, as a function of temperature and pressure. The filled circles mean that hematite was converted to goethite, or that goethite was not converted to hematite, and the open circles mean that goethite was converted to hematite, or that hematite was not converted to goethite. The other conditions are described in Table 2, to which the experiment numbers refer.

solution, the decomposition temperature is  $165^{\circ} \pm 5^{\circ}$  C. Allowing for the fact that the decomposition point is difficult to locate near neutrality due to the great insolubility of iron oxide in water, the best data has been plotted in Fig. 1. This gives the approximate decomposition temperature of goethite at a low pressure as a function of acidity-alkalinity. Probably the limits of  $\pm 0.1M$  free acid and alkali enclose most of the conditions in hydrothermal solutions, and therefore limit the range of the decomposition point at low pressures in nature.

The effect of pressures up to 2000 atmospheres on the decomposition temperature of goethite in neutral and alkaline solutions is of a small order. The decomposition temperature is raised less than  $5^{\circ}$  C. per 1000 atmospheres pressure in 0.1M NaOH solutions. The experimental points

and the phase boundary in the alkaline solution are shown in Fig. 2.

Since the decomposition temperature of goethite is virtually insensitive to pressures as high as those which would be found in the earth where temperatures near the decomposition point prevail, the goethite-hematite relation can be used in geological thermometry. Normal hydrothermal solutions stable with the rock forming minerals must be neutral to weakly alkaline, so that the decomposition temperature of goethite may be taken with some assurance to be  $150^{\circ} \pm 20^{\circ}$  C. in hydrothermal solutions. If such solutions deposit ferric oxide above this temperature, it will be as hematite, and below, as goethite. The stable field of the rare mineral lepidocrocite, is not yet defined.

#### ACKNOWLEDGMENTS

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# A DECREPITATION GEOTHERMOMETER

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## ABSTRACT

In the past the use of fluid inclusions in minerals as a geothermometer has been confined to the direct observation of the disappearance of the gas phase by means of a microscope and heating stage. More recently the breaking out of the inclusions as the mineral is heated, has served to indicate the temperature of complete filling of the inclusions by the liquid phase. Apparatus is described for recording the rate of break out and the interpretation of the recorded curves is discussed.

Fluid inclusions in hydrothermal minerals may usually be readily observed, for they range in size from the limits of resolution of the microscope to several centimeters in length. They are in general of two types: (1) primary inclusions formed at the time of the initial crystallization of the mineral, and (2) secondary inclusions formed subsequently, by recrystallization in fractures (Newhouse, 1932).

Sorby (1858) suggested that the fluid existed in the inclusions at the time of its formation as a single phase and that on cooling from the crystallization temperature a second, gaseous phase appeared due to the contraction of the liquid. He showed that the temperature at which the gas phase appears during cooling is determined by the temperature, pressure, and composition of the liquid at the time of being trapped. Holden (1925), Newhouse (1933), Twenhofel (1947), Ingerson (1947), and others have used this idea of Sorby's to obtain information regarding the temperature and pressure of formation of minerals. The method used by them was to heat a fragment of crystal and watch, by means of a microscope, the change in size of the gas bubble in the inclusions. Thus the temperature at which the gas phase disappeared, that is, the filling temperature, was determined. From this, with the aid of specific volume/temperature/pressure curves, the specific volume of the liquid under the conditions of formation was calculated.

## RECENT WORK

The disadvantages of the heating stage-microscope method are obvious and have been outlined by Scott (1948) in a recent paper. The technique developed by Scott entails heating a sample of the mineral under study in a glass tube, listening to the progress of the decrepitation by means of a stethoscope or microphone-audio amplifier link and interpreting what is heard in the form of manuscript notes. The sound produced has been likened to the bubbles bursting in a newly opened bottle

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of carbonated water. It is produced by the breaking out of the liquid from the inclusions as a result of the very sudden increase in pressure as the mineral is heated above the filling temperature. This break out results in a true explosion, since at temperatures above the boiling point of the liquid, water vapour is generated very quickly. Hence even very small inclusions produced definite "pops." A modification of the above technique is currently in use in this department, wherein the course of the decrepitation is automatically recorded.

#### FEATURES OF DECREPITATION

In making a record of the progress of decrepitation the following variables may be determined:

- (a) the loudness of the popping,
- (b) the temperature at which the first pop occurs,
- (c) the frequency of the popping.

The loudness of the explosions is controlled by the size of the inclusion and speed with which it can break out. It is not necessarily controlled by the temperature of formation.

The temperature at which the first pop is heard is theoretically the temperature at which complete filling just takes place. However this will only be true if the layer of mineral lying between the inclusion and the surface is strong enough to withstand the vapour pressure of the liquid at temperatures below that of complete filling. If not, the inclusion will pop at a temperature below the filling temperature. Furthermore if there are few inclusions it may be possible that none lies in the ideal position for break out as complete filling is reached, namely, just beneath the surface. In such a case the appearance of the first pop will be delayed.

The frequency of the pops is a function of the number of inclusions, their relative closeness to the surface, the degree of comminution of the particles, the rate of rise of temperature and the ratio of the pressure in the inclusions to the strength of the inclusion walls. It is possible for an inclusion to lie at all possible depths below the surface. Those lying closest to the surface will pop at a temperature nearest to that at which the cavity is just filled, followed in turn by the inclusions more and more deep seated, as greater pressure is required to break them out. By plotting frequency against temperature a curve will be produced which will approximate that of the rise in pressure inside a deep-seated inclusion. The inflection point in such a curve will then be the point at which the cavity is just filled by the expanding liquid, allowance being made for possible overshoot due to the strength of the mineral in tension.

It was found that, while frequency/temperature curves could be drawn from the manuscript notes made when listening to the decrepita-



tion, a more precise method was necessary. Apparatus was therefore designed to listen to the decrepitation and simultaneously plot the frequency against temperature.

#### DESCRIPTION OF THE APPARATUS

The equipment consists essentially of the following units. (a) *A means of heating the sample evenly and with a constant rate of increase in temperature.* This may be achieved by heating the sample in a "Pyrex" glass tube placed at the centre of a small electric muffle furnace. A thermocouple wired to a temperature indicating device is inserted in a small dimple in the tube, care being taken to ensure its lying as close as possible to the charge. The temperature rise is controlled by manipulation of a variable transformer and should be of the order of 15° C. per minute.

(b) *A pressure type crystal microphone coupled to the "Pyrex" glass tube.* Due to the very high gain of the amplifier fed by the microphone, care has to be exercised in shielding the latter from building and room noises. This was achieved here by enclosing the microphone in a series of boxes, one inside another, the space of about one inch between them being packed loosely with cotton wool.

(c) *A simple high gain audio amplifier to amplify the output of the microphone.* Filters can be incorporated at this point to cut out rumble not removed by the microphone shielding. Provision should be made to monitor the output of the amplifier by means of earphones.

(d) *An electronic relay of the unbalanced multivibrator type.* This converts the pulses (produced by the pops) of varying amplitude and duration, into pulses of constant value. The recovery time of the relay is made variable so that full scale deflection on the final recording meter might be made to correspond to 0.1, 1.0, 10 and 100 pops per second.

(e) *A resistance/capacity integrating circuit.* This integrates the pulses as received from the relay and delivers a voltage which is proportional in value to the frequency of the pops. This voltage controls a direct coupled power amplifier.

(f) *A recording milliammeter.* This then draws a curve of frequency of decrepitation against time (so-called "decrepigraph"). Temperature fiducials are superimposed on the curve by operating a push button at 20°C. intervals read from a temperature indicator.

A diagram of the relation between the components of the apparatus is shown in Fig. 1, and a photograph of the apparatus in use is shown in Fig. 2.

#### PREPARATION OF THE CHARGE

A sample of the mineral under study is crushed in a mortar and the particles are sized, although for a preliminary run it may be advantageous

to have unsized fragments. The optimum size of particles is controlled by a number of factors, the most important being the size and distribution of the inclusions and the strength of the mineral in tension. Obviously if the inclusions are large in size and few in number the sample should not be crushed fine enough to destroy them. It has been found, however, that where the inclusions range from very large to very small in a single sample there is a tendency for the large ones to break out before the point of complete filling, followed immediately by a sudden rain of smaller in-

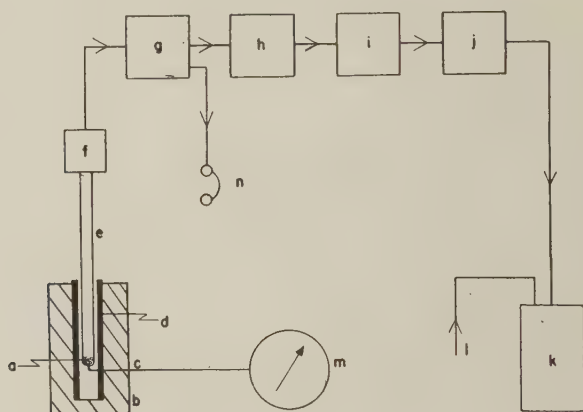


FIG. 1. Diagram of apparatus. Electronic section shown in block form. (a) Sample of material, (b) furnace insulation, (c) thermocouple, (d) furnace heating element, (e) glass tube, (f) microphone, (g) audio amplifier, (h) electronic relay, (i) integrating circuit, (j) power amplifier, (k) recording milliammeter, (l) input of temperature fiducials, (m) temperature indicator, (n) monitoring earphones.

clusions breaking into the cavity so formed. This produces an anomaly in the decrepitation curve. Under such circumstances it is better to make a series of runs, each with particles of a different size, until the anomalies disappear. Care should be taken to ensure as clean a charge as possible. Fragments of minerals other than that under study will result in a curve which is multiple. Treatment with acid is recommended, particularly if there is indication of the presence of carbonate minerals. The carbonates have a tendency to crystallize in fractures, trapping secondary inclusions which may bear no relationship to those in the mineral being studied. After such treatment the sample should be very well washed (to prevent vaporized acid damaging the microphone), and thoroughly dried. Drying is essential, since water trapped between fragments and in fractures produces a noise similar to decrepitation when the boiling point of water is exceeded.

## RECORDING DECREPIGRAPHS

The curves drawn by the recorder represent frequency of decrepitation against time. The time/temperature heating curve may be drawn on the same time base using the temperature fiducials. Thus the temperature of any point on the decrepitation curve can readily be found.

The apparatus has been provided with a number of controls, the purpose of which is to introduce flexibility into the system. In addition, there



FIG. 2. Photograph of the apparatus in use. (a) Small electric muffle furnace, (b) glass tube carrying the charge, (c) microphone in sound insulating box, (d) audio amplifier, (e) electronic relay, integrating circuit, and power amplifier (f) temperature indicator, (g) variable transformer for controlling the furnace, (h) recording milliammeter, (i) monitoring earphones, (j) push switch for producing temperature fiducials on the curve.

are the variables of size and number of inclusions and of the grain size of the sample. Varying any one of these has a bearing on the shape of the final curve.

Since the size of the inclusions governs the loudness of the pops and since the apparatus has a definite threshold above which it will hear the pops, the size of the inclusions will determine whether they are recorded or not. A gain control has been provided on the amplifier to compensate for this, manipulation of which will produce an effect which is the reciprocal of a change in the size of inclusions.

The number of inclusions present in a sample, whether due to the size of the sample or due to the number of inclusions per unit volume of material, will govern the frequency of decrepitation, since, if there are fewer inclusions present there will be a smaller number available to break out per unit increment of temperature rise. A control on the electronic relay changes the counting rate to compensate for differences in the frequency of popping from sample to sample. This control however may cause difficulty in the correct interpretation of the curves if its effect is not fully understood. The use of too high a counting rate may cause the curve to be

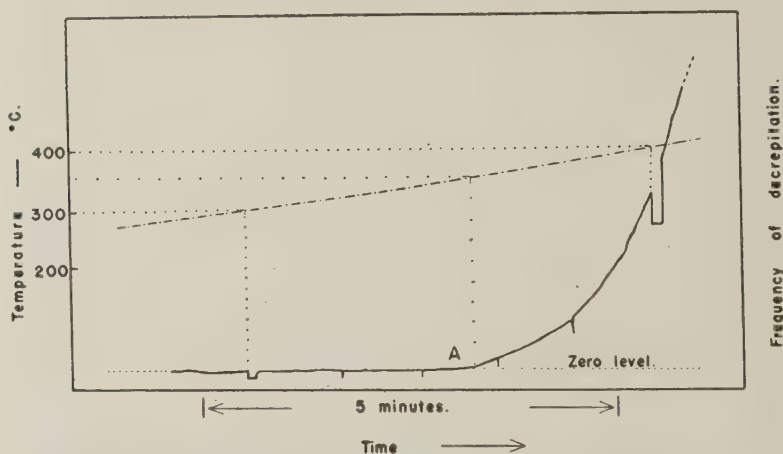


FIG. 3. Part of decrepigraph of artificial quartz. Particles sized  $-40 +60$  mesh. Solid line frequency/time. Broken line temperature/time on same time base. Point A corresponds to  $355^{\circ}\text{C}$ .

flattened to such an extent that the inflection point cannot be readily found. This may result in a temperature reading which is too high. If the counting rate is too low, spurious popping may drive the recorder full scale and an anomalous curve result. Experience plus information from a preliminary run will best indicate the correct setting of the range control.

It was found desirable to be able to vary the time interval over which the frequency of decrepitation be integrated. Therefore a control is provided giving a choice of long, medium, or short integration time, the effect of which is to tend to smooth out any irregularities in the curve, the greatest smoothing being in the position of longest integration time. However the use of a long integrating time when making a decrepigraph will have the tendency to round off also any sudden changes in decrepitation frequency and may make the inflection point difficult to determine. Therefore the shortest integration time should be chosen, consistent with the frequency of decrepitation and the drawing of a smooth curve.



## INTERPRETATION OF DECREPIGRAPHS

In the majority of cases the curves have a form similar to that in Fig. 3, with an inflection point as at point "A" where the rate of decrepitation increases very suddenly. This point can be taken to represent the temperature of complete filling of the inclusions with an allowance for possible overshoot. The filling temperature may then be applied to a set of curves such as those shown by Ingerson (1947), Béland (1948), or Scott (1949) and the temperature/pressure relation found.

Fig. 3 is a decrepigraph of some artificial quartz grown by the Brush Development Company in a dilute sodium carbonate solution having, at the temperature of formation, a specific volume of  $1.53 \text{ cm}^3/\text{gm}$ . The temperature of complete filling is therefore near  $340^\circ \text{C}$ . As will be seen from the decrepigraph, the inflection point is at  $355^\circ \text{C}$ ., indicating an overshoot of  $15^\circ \text{C}$ .

The sample used in the above run was crushed in a mortar and sized at  $-40 +60$  mesh. Comparison of Fig. 3 with Fig. 4 shows the effect, mentioned above, of change in particle size. The material used to produce the curve in Fig. 4 was similar to that for the previous curve except that it consisted of random sized particles ranging from  $\frac{1}{4}$  inch diameter, downwards. The small anomaly in Fig. 4 is the result of a number of large inclusions breaking out at about  $330^\circ \text{C}$ . (due probably to the vapour pressure), followed by a rain of small inclusions possibly leaking out along newly formed fractures. This produces a hump in the curve which hides the true inflection point. In some cases the decrepitation of second-

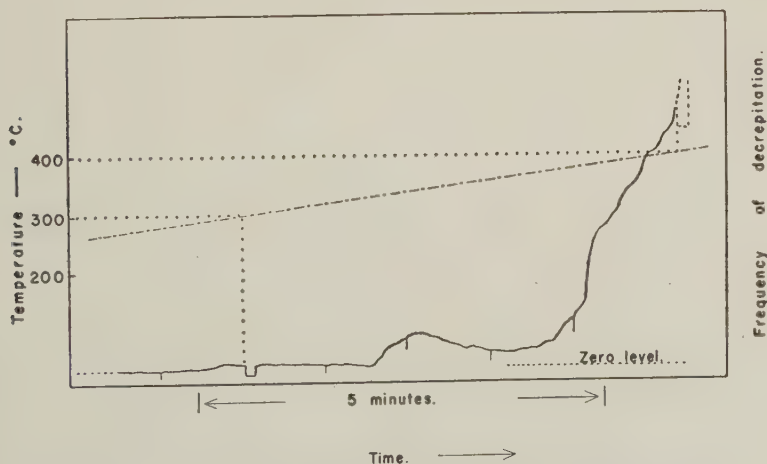


FIG. 4. Part of decrepigraph of same artificial quartz as in Fig. 3. Particles unsized, ranging from  $\frac{1}{4}$  inch diameter downwards.

ary inclusions, formed at a temperature below that of the mineral studied, will add to the decrepitation of the primary inclusions. The curve so produced will be the resultant of two curves, the resolution of which may be carried out mathematically or graphically. In practice however, it is found sufficient to extrapolate the front of the main decrepitation curve back, until it intersects the zero level base line, and the point so found will be approximately the inflection point sought. If the secondary inclusions begin to explode at a temperature only slightly below that of the primary inclusions, the two series can be detected readily by inspection of the decrepigraph, but an exact separation is difficult.

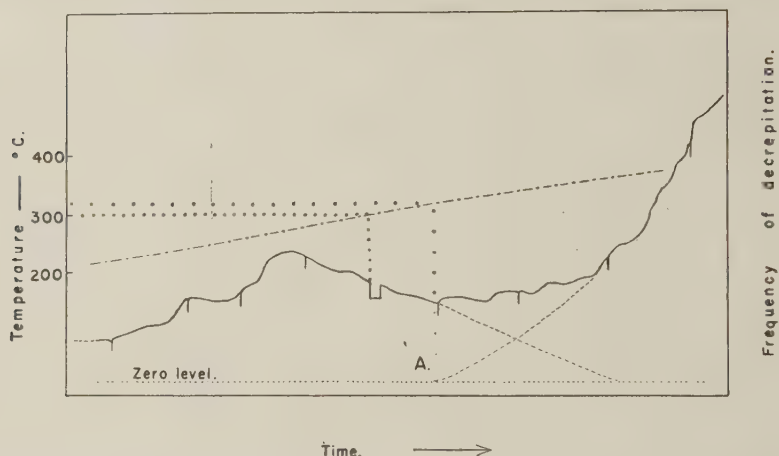


FIG. 5. Part of decrepigraph of sphalerite from Kansas lead/zinc deposit. Inflection point hidden by decrepitation of secondary inclusions. Curves extrapolated to zero level. Point A is the inflection point on primary inclusion curve.

Fig. 5 is an example of a decrepigraph showing the effect of secondary inclusions (in sphalerite from the Kansas-Oklahoma portion of the Tri-State lead-zinc deposits) which add to, but do not obscure, the effect of the primary inclusions. The probable positions of the two simple curves have been drawn.

While the data from decrepitation curves may be used to obtain the temperature/pressure conditions of deposition of a mineral, they are also useful in providing information about the paragenesis of minerals in a deposit. Assuming that the minerals are deposited in order of decreasing temperature, a series of decrepigraphs will show the order of crystallization. This will be true only if the pressure over the whole range of deposition remains unchanged, a not unreasonable assumption in most cases, since it is dependent almost exclusively on the "hydrostatic" pressure of

the overlying rock and this is not likely to change greatly during the formation of a deposit.

It has been found that practically all of the minerals in deposits believed to be of hydrothermal origin contain an abundance of liquid inclusions, and give resolvable decrepigraphs. However, a number of results have been obtained which are at variance with accepted temperatures of formation of some deposits. Taking into account that many of such accepted temperatures of deposition are based on opinions rather than on measurements, it is possible that the decrepitation method will allow a new scale of such temperatures to be prepared. Deposits of this type, where the decrepitation measurements indicate temperatures of deposition much different from the commonly accepted values include the Lake Superior hematite deposits, the Tri-State lead-zinc deposits, some of the gold-quartz vein deposits in the Canadian Shield, and the later minerals in certain pegmatite dikes. Ingerson (1947) has pointed out that low temperature deposition of quartz in pegmatites is not unusual. Investigations are in progress and much work has yet to be done on these deposits using the decrepitation method before any general conclusions can be drawn.

#### ACKNOWLEDGMENTS

To Dr. F. G. Smith of this department, who was responsible for suggesting much of the technique and who shared in the design of the equipment, the writer wishes to express his thanks for much encouragement and helpful criticism. Appreciation is also expressed to Dr. D. R. Hale of the Brush Development Company, Cleveland Ohio, who kindly provided the artificial quartz.

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# CHLORITOID FROM MEGANTIC COUNTY, QUEBEC<sup>1</sup>

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## ABSTRACT

Chloritoid occurs as greenish black plates (up to  $1 \times 20$  mm.) in quartz-carbonate veins, at the Harvey Hill mine, 5 miles E. of Leeds, Megantic County, Quebec. Cleavage (001) good, (*hkl*) poor.  $H = 6\frac{1}{2}$ .  $G = 3.528$ . The monoclinic unit cell with space-group  $C2/c$ , has  $a = 9.45$ ,  $b = 5.48$ ,  $c = 18.16$  Å,  $\beta = 101^\circ 30'$ , and contains  $4[(Fe, Mg)_2Al_2(Al_2Si_2)O_{10}(OH)_4]$ . Twinning lamellar, probably with twin axis [130], composition plane (001). Optically positive;  $X = b$ ,  $Z:(001) = +15^\circ$ ,  $Z:[001] = +26\frac{1}{2}^\circ$ .  $\alpha$  (greenish grey to olive green)  $= 1.716$ ,  $\beta$  (bluish grey to idigo)  $= 1.719$ ,  $\gamma$  (colourless to greenish yellow)  $= 1.725$  (all  $\pm 0.001$  for Na);  $2V = 66 \pm 2^\circ$ ,  $r > v$ .

Early crystal measurements indicate the forms  $c(001)$ ,  $m(110)$ ,  $e(401)$ ,  $i(\bar{4}01)$ ,  $k(114)$ ,  $h(\bar{1}11)$ ; an angle-table for these forms is given on  $a:b:c = 1.7245:1:3.3141$ ,  $\beta = 101^\circ 30'$  (x-ray). The powder pattern of chloritoid is indexed and a review of 20 analyses establishes the composition  $(Fe'', Mg)_2(Al, Fe''')(Al_2Si_2)O_{10}(OH)_4$ .

Chloritoid was named by G. Rose (1837, in Dana, 1892, p. 640) from its superficial resemblance to chlorite, and the mineral is generally placed in the Clintonite Group or Brittle Micas which are chemically intermediate between the true micas and the chlorites. The brittle micas resemble the true micas in crystal form, cleavage, and optical properties, but they differ in the brittleness of their cleavage plates. Chloritoid is particularly distinguished by its difficult basal cleavage and high hardness ( $6\frac{1}{2}$ ). Analyses of chloritoid and also the optical and physical properties vary somewhat; this led to the introduction of such names as barytophyllite, masonite, sismondine, struverite, and salmite, which are included under chloritoid by Dana. The empirical formula given by Dana (1892) is  $H_2(Fe, Mg)Al_2SiO_7$ ; and later work, by Simpson (1931) and by Balk and Barth (1934) has confirmed these proportions, when allowance was made for inclusions of quartz and rutile.

Although chloritoid has been studied many times in the past, its properties are still not completely described. A specimen from the Harvey Hill Mine in Megantic County, Quebec (Royal Ontario Museum M6366) provided sharply crystallized material for a full examination. Similar material was described and analysed by Hunt (1861) whose analysis has been used in the calculation of the cell content.

## GEOLOGY AND PETROGRAPHY

The Harvey Hill Mine, 5 miles E. of Leeds, Megantic County, was a copper prospect and producer from approximately 1850 to 1900. According to Bancroft (1915) the mine is located in slaty schists of Precambrian

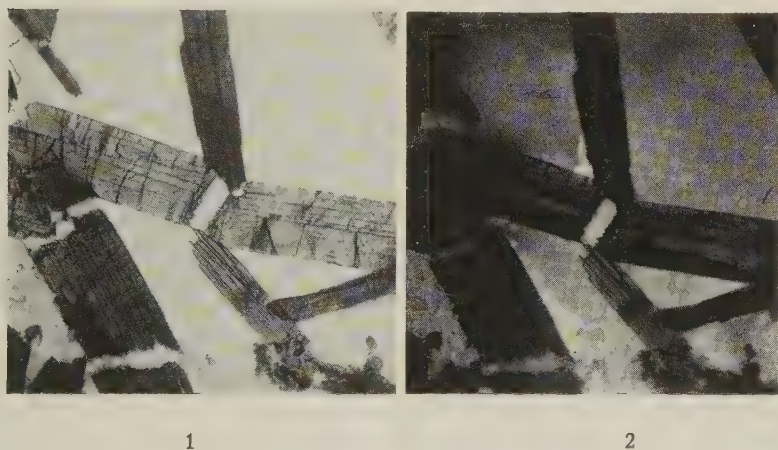
<sup>1</sup> Awarded the Walker Mineralogical Club prize for 1948.

<sup>2</sup> Graduate student and class assistant, Department of Geological Sciences.



age; some bands in these schists are rich in chloritoid. Veins composed of quartz, calcite, and dolomite traverse the schists and it seems likely that the museum specimen came from one of these veins, for it is composed largely of quartz and carbonate with a small amount of light grey-green wall-rock.

The chloritoid is most abundant in the quartz-carbonate vein with a smaller amount in the wall-rock. It occurs in greenish black plates up to 1 mm. thick and 20 mm. wide, rarely showing roughly hexagonal outlines and more often traversed by quartz-filled fractures intersecting at about  $60^\circ$ . A thin section from the vein portion of the specimen (Figs. 1, 2) shows plates of chloritoid embedded in coarse quartz, with smaller



FIGS. 1, 2. Chloritoid, Megantic County, Quebec; thin section showing basal plates on edge in quartz-carbonate vein;  $\times 13$ . Fig. 1 (left). One nicol. Fig. 2 (right). Crossed nicols, showing lamellar twinning.

quantities of calcite, apatite, and white mica. The plates are sometimes fractured and recemented by finer grained quartz. These chloritoid plates are otherwise quite free from inclusions. A similar occurrence of chloritoid, from Crestlianderstobel in the Swiss Alps, has been described by Friedlaender (1930). Here the chloritoid occurs in a schistose rock and contains inclusions of quartz, but small quartz veins traversing the rock contain aggregates of the mineral free of inclusions.

#### PHYSICAL PROPERTIES

The large greenish black plates of chloritoid are almost opaque, but small fragments are transparent and bottle-green in colour. There is a perfect but difficult cleavage parallel to (001), and the lamellae break easily roughly perpendicular to this plane. Fragments broken in this way

are often quite irregular in outline, but occasionally there are two distinct breaks intersecting at  $60^\circ$ . The faces produced are very imperfect and give a diffuse train of signals on the optical goniometer. X-ray work proved that these poor cleavages are of the type (*hhl*) (Fig. 3).

The lustre is vitreous to brilliant on the (001) face, but subvitreous on irregular surfaces. The powder is grey-green in colour. Careful hardness measurements placed the mineral between feldspar and quartz; this value of  $6\frac{1}{2}$  is the upper limit which has been reported for chloritoid by previous investigators. The remarkable hardness distinguishes chloritoid from other members of the Brittle-Mica Group and suggests that it may not properly belong in this group.

Specific gravity measurements were made using Clerici's solution and the Westphal balance, from which an average value of 3.528 was obtained. This is in close agreement with the value 3.513 reported by Hunt for chloritoid from Megantic County.

#### CRYSTAL FORM

Plates of chloritoid from Megantic County generally have ragged edges, but occasionally a rough hexagonal outline can be detected. Faces other than (001) are lacking and the latter is often curved and not parallel to its opposite face. In several cases crystals were elongated in the [110] direction, as proved by a series of rotation and Weissenberg x-ray photographs.

Tschemmak and Sipöcz (1879) were able to measure the interfacial angles between *c*(001) and five other faces on chloritoid from Pregratten, Tyrol. Since this is the only record of such measurements, an attempt has been made to correlate Tschemmak's measured angles with angles calculated from the cell elements. A tentative and not wholly satisfactory correlation of forms and angles is given below.

Tschemmak	Measured	Milne	Calculated
<i>cm</i> = (001):(332)	$83^\circ 25'$	<i>cm</i> = (001):(110)	$84^\circ 10\frac{1}{4}'$
<i>ce</i> = (001):(011)	71	<i>ce</i> = (001):(401)	71 25
<i>cj</i> = (001):(061)	86 30	<i>cj</i> = (001):(40 $\bar{1}$ )	85 $57\frac{1}{2}$
<i>cn</i> = (001):(111)	86 06	<i>cn</i> = (001):(111)	80 40
<i>ck</i> = (001): —	40 ca.	<i>ck</i> = (001):(114)	40 $34\frac{1}{2}$

Table 1 gives a formal angle-table for the forms indicated by Tschemmak's measurements, with reference to the geometrical elements of the crystal lattice determined later by x-rays.

Polysynthetic twinning is universal, with (001) as the composition face (Fig. 2). Consideration of the new cell dimensions indicates the probability of twinning according to the law: twin axis [130], composition plane (001). The basal planes on the two individuals would be brought

TABLE 1. CHLORITOID:  $(\text{Fe, Mg})_2\text{Al}_2(\text{Al}_2\text{Si}_2)\text{O}_{10}(\text{OH})_4$ Monoclinic,  $C2/c$  $a:b:c=1.7245:1:3.3141$ ;  $\beta=101^\circ 30'$  $p_0:q_0:r_0=0.5918:1:0.3079$ ;  $\mu=78^\circ 30'$  $p'_0=1.9611$ ,  $q'_0=3.3141$ ,  $x'_0=0.2134$ 

Form	$\phi$	$\rho$	$\phi_2$	$\rho_2=B$	C	A
<i>c</i> (001)	90° 00'	11° 30'	78° 30'	90° 00'	0° 00'	78° 30'
<i>m</i> (110)	30 37	90 00	0 00	30 37	84 10	59 23
<i>e</i> (401)	90 00	82 55	7 05	90 00	71 25	7 05
<i>j</i> ( $\bar{4}$ 01)	-90 00	82 32	172 32½	90 00	94 02½	172 32½
<i>k</i> (114)	39 56½	47 13	55 15	55 45½	40 34½	64 09
<i>h</i> (111)	-27 56½	75 04½	15 22	31 23½	80 40	96 56

into exact parallelism and the zone axes [100], [110], [010], on one individual would fall very nearly over the zone axes [130], [010], [110], respectively, on the other individual. This is comparable with the twinning in muscovite described by Peacock and Ferguson (1943), except that in muscovite the *a* and *b* axes are interchanged as compared to chloritoid, and hence the twin axis is [310].

## OPTICAL PROPERTIES

The acute positive bisectrix *Z* is inclined at  $15^\circ$  to the normal to (001), as measured with the universal stage on the same crystal which was later used for x-ray work. It will be seen in Table 2 that the positive sign of chloritoid is well established, with *Z* inclined to the normal to (001) at angles varying from  $5^\circ$  to  $30^\circ$ . Correlation of optical observations and x-ray measurements on a single crystal from Megantic County showed that *X* corresponds to the symmetry axis *b*; this does not agree with the observations of Balk and Barth, who reported that *Y* was always contained in the cleavage plane (001) and presumably is therefore coincident with *b*. The inclination of *Z* to the *c*-axis is  $Z:c=+26\frac{1}{2}^\circ$  and hence  $Y:c=-63\frac{1}{2}^\circ$ .

Fragments of the mineral approximately 0.25 mms. in thickness exhibit a remarkable pleochroism, *X* greenish grey to olive green, *Y* bluish grey to indigo, and *Z* colourless to greenish yellow, giving the absorption formula,  $Y>X>Z$ . All past observers have noted this relationship, the average colours being *X* green, *Y* blue, *Z* pale yellow.

The indices  $\alpha$  and  $\beta$  were obtained by immersion, with sodium light, from flat-lying cleavage flakes. Because of the small inclination of *Y* to the cleavage plane (001) the error in  $\beta$  is negligible. The value of  $\gamma$  was obtained from fragments lying perpendicular to *c*(001) and giving the

maximum extinction angle ( $15^\circ$ ). The refractive indices (Na) are:

$$\alpha=1.716, \beta=1.719, \gamma=1.725, \text{ all } \pm 0.001$$

Observations by other authors give values for  $\beta$  ranging from 1.719 to 1.728, with an average birefringence of 0.009.

The optic axial angle,  $2V$ , was obtained from the apparent optic axial angle in glass,  $2G$ , measured with sodium light on the universal stage, using glass hemispheres of refractive index  $n$ . The angle  $V$  is related to the angle  $G$  by the relationship,  $\sin V = \sin G \cdot n/\beta$ .  $2V$  may be calculated from the refractive indices using the approximate formula  $\cos^2 V = (\beta - \alpha)/(\gamma - \alpha)$ . The values obtained by the two methods are:

$$2V = 66 \pm 2^\circ \text{ (measured), } 66^\circ \text{ (calculated)}$$

Previously reported measurements of  $2V$  are mostly in the range  $50^\circ - 68^\circ$ . The dispersion is perceptible,  $r > v$ . This agrees with other observations recorded in Table 2, except that Balk and Barth (1934) reported the dispersion of Dutchess County chloritoid to be anomalous, with green on the convex side of the hyperbolas and yellow on the concave side.

TABLE 2. CHLORITOID: OPTICAL OBSERVATIONS

	1	2	3	4	5	6	7
$\alpha$ .....	1.716	1.715	1.720	1.722	1.724	1.724	—
$\beta$ .....	1.719	1.719	1.722	1.725	1.726	1.726	1.728
$\gamma$ .....	1.725	1.737	1.731	1.728	1.730	1.737	—
$\gamma - \alpha$ .....	0.009	0.022	0.011	0.006	0.006	0.013	0.007
$X$ .....	Greenish grey to olive green	Olive to olive green	Colourless to pale gn. yellow	Pale grey green	Greenish blue	Greenish brown to grass green	Yellowish green
$Y$ .....	Blue grey to indigo	Blue	Plum to indigo blue	Slate blue	Blue	Deep blue to bluish green	Bluish green
$Z$ .....	Colourless to greenish yellow	Pale	Yellow to colourless	Yellow to colourless	Pale yellow	Yellowish green	Pale yell. to colourless
$X=b$ .....	—	—	—	$Y=b$	—	—	—
$Z:(001)$ .....	$15^\circ$	—	$20^\circ$	$5^\circ - 22^\circ$	$30^\circ$	$21^\circ$	$15^\circ$
Sign.....	+	—	+	+	+	—	+
$2V$ .....	$66^\circ$	$50^\circ$	$36^\circ - 60^\circ$	$60^\circ$	$68^\circ$	$63^\circ$	—
Disp.....	$r > v$	—	$r > v$	Anomalous	$r > v$	—	—

1. Megantic Co., Que. (anal. 13, Table 4); Milne (this paper).
2. Saas Valley, Switzerland (anal. 3); Friedlaender (1930).
3. Locality unknown; Larsen and Berman (1934, p. 131).
4. Dutchess Co., N.Y. (anal. 19); Balk and Barth (1934).
5. Champion Mine, Michigan (anal. 11); Milne (this paper).
6. Crestlianderstobel, Switzerland (anal. 8); Friedlaender (1930).
7. Deep River Region, N.C. (anal. 20); Stucky (1926).

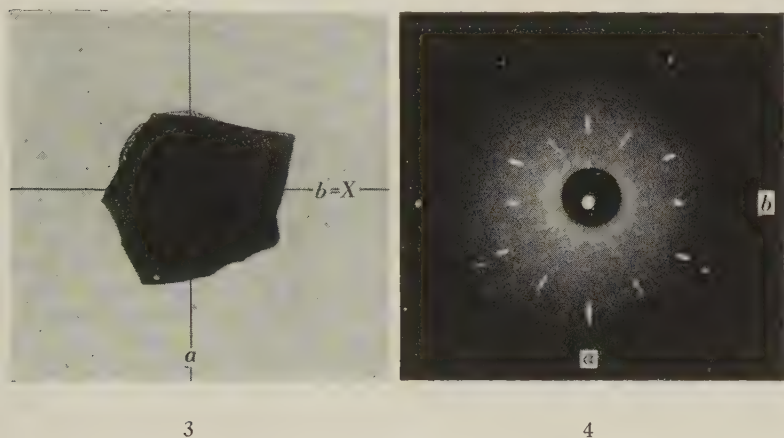
#### STRUCTURAL CRYSTALLOGRAPHY

A thin cleavage flake of chloritoid (Fig. 3), free from twinning and showing evidence of two cleavage directions intersecting at  $60^\circ$  in the



basal plane, was selected for x-ray measurements. The plane containing  $Y$  and  $Z$  bisects the angle between the two cleavage traces. One edge of the plate, which appeared to be due to such a cleavage, gave a train of poor signals on the optical goniometer; individual signals could not be measured but the zone was well defined.

A basal Laue photograph (Fig. 4) was taken of this crystal using tungsten radiation. The diffraction pattern indicates a plane of symmetry, with the optical direction  $X$  coinciding with the perpendicular to this plane.  $Y$  and  $Z$  lie in the plane of symmetry.



FIGS. 3, 4. Chloritoid, Megantic County, Quebec. Untwinned single crystal plate used for x-ray photographs, with traces of poor ( $hhl$ ) cleavage. The directions of the axes  $a$  and  $b=X$  are indicated. Fig. 3 (left). One nicol;  $\times 20$ . Fig. 4 (right). Laue photograph showing plane of symmetry perpendicular to  $b=X$ .

The crystal was then set up for rotation about the symmetry axis ( $X$  direction) and a rotation photograph and zero, first and second layer Weissenberg resolutions were taken. These photographs showed that the symmetry is monoclinic; the smallest and least oblique cell has the dimensions:<sup>3</sup>

$$a=9.45, b=5.48, c=18.16 \text{ \AA}, \beta=101^\circ/30'$$

The systematically missing spectra, namely ( $hkl$ ) present only with ( $h+k$ ) even, ( $h0l$ ) present only with  $h$  even and  $l$  even lead to the space-groups,  $Cc$  or  $C2/c$ .

The only published work on the cell dimensions of chloritoid is by Machatschki and Mussnug (1942). These authors reported the following values:

$$a=5.4, b=9.4, d(001)=17.85 \text{ \AA}$$

<sup>3</sup> Using  $\lambda \text{ CuK}\alpha_1=1.5418 \text{ \AA}$ .

The measurements of  $a$  and  $b$  are obviously rough and apparently interchanged, and no attempt was made to measure  $\beta$ . The value for  $d(001)$  compares with that obtained during this investigation, namely 17.80 Å.

Before the discovery of the exceptional crystal used in the above cell determination, a series of x-ray photographs of a log-shaped cleavage fragment had been taken. The fragment showed a good basal cleavage and, as was proved later, a very poor cleavage ( $hkl$ ). Optical results were indefinite but the extinction was distinctly inclined suggesting triclinic symmetry. The resulting rotation and Weissenberg photographs gave a unit cell with the dimensions:

$$a' = 9.49, b' = 5.45, c' = 17.98 \text{ Å} \\ \alpha' \sim 90^\circ, \beta' = 98^\circ 51', \gamma' \sim 90^\circ$$

A second-layer Weissenberg resolution indicated that  $\alpha'$  and  $\gamma'$  are not exactly  $90^\circ$ , but it was impossible to measure the exact angles.

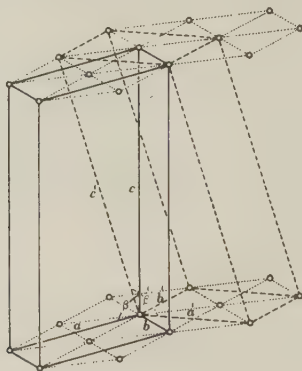


FIG. 5. Chloritoid. Crystal lattice showing the true monoclinic cell  $a b c \beta$ , and false triclinic cell  $a' b' c' \beta'$ .

When the true monoclinic cell was finally determined, it was realized that there is present in the structure a false triclinic cell in which  $\alpha'$  and  $\gamma'$  are close to  $90^\circ$  and  $a', b', c'$  are near to those of the true cell. The relationship between these cells is shown in Fig. 5. In order to check this relationship, the dimensions of the false triclinic cell ( $T$ ) have been calculated from those of the true monoclinic cell ( $M$ ).

True Cell ( $M$ )	False Cell ( $T$ )	
Measured	Measured	Calculated
$a = 9.45 \text{ Å}$	$a' = 9.49 \text{ Å}$	9.48 Å
$b = 5.48$	$b' = 5.45$	5.46
$c = 18.16$	$c' = 17.98$	18.04
—	$\alpha' = 90^\circ$	$88^\circ 39'$
$\beta = 101^\circ 30'$	$\beta' = 98^\circ 51'$	$99^\circ 20'$
—	$\gamma' = 90^\circ$	$89^\circ 45'$

Structural studies of the micas have shown that the cleavage plane in these minerals is parallel to hexagonal layers with  $a=5.2\text{--}5.3$ ,  $b=9.0\text{--}9.2$ , and a vertical spacing of nearly 10.0 Å between successive layers. The two-layer type (muscovite), is the most closely related in geometrical properties to chloritoid, but there are considerable differences, as shown below.

Chloritoid	Muscovite
$C2/c$	$C2/c$
$a=9.45$ Å	5.21 Å
$b=5.48$	9.02
$c=18.16$	19.98
$\beta=101^\circ 30'$	$96^\circ 24'$

A powder photograph of the material from Megantic County was taken with Fe radiation and a Mn filter. The measured spacings have been indexed and they are compared with calculated spacings in Table 3. So far as is known, these are the first x-ray powder data to be given for chloritoid.

TABLE 3. CHLORITOID: X-RAY POWDER PATTERN

Monoclinic  $C2/c$  $a=9.45$  Å,  $b=5.48$ ,  $c=18.16$  Å,  $\beta=101^\circ 30'$ 

$I$	$\theta(\text{Fe})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$	$I$	$\theta(\text{Fe})$	$d(\text{meas.})$	$(hkl)$	$d(\text{calc.})$
10	12.55°	4.46	{(004)	4.45	$\frac{1}{2}$	29.22°	1.983	(315)	1.986
			{(111)	4.45	1	30.92	1.884	(318)	1.882
$\frac{1}{2}$	16.60	3.39	{(114)	3.41				{(130)	1.793
3	19.08	2.96	(006)	2.97	$\frac{1}{2}$	32.68	1.794	{(408)	1.793
1	20.60	2.75	(020)	2.74				{(510)	1.755
			{(116)	2.63	$\frac{1}{2}$	33.60	1.749	{(319)	1.744
2	21.65	2.62	{(313)	2.63				{(515)	1.706
			{(022)	2.62	$\frac{1}{2}$	34.58	1.706	{(317)	1.705
1	22.70	2.51	{(314)	2.50	$\frac{1}{2}$	35.92	1.650	{(516)	1.653
1	23.20	2.46	(312)	2.46				{(135)	1.577
			{(402)	2.36	3	37.70	1.781	{(1.1.11)	1.580
3	24.22	2.36	{(220)	2.36				(424)	1.563
			{(315)	2.35				(136)	1.561
			{(400)	2.31	1	38.30	1.562	(2.0.10)	1.560
3	24.88	2.30	{(313)	2.30				{(333)	1.560
			{(206)	2.30				{(3.1.12)	1.406
$\frac{1}{2}$	25.50	2.25	(404)	2.24	1	43.48	1.407	{(608)	1.405
1	27.02	2.13	(402)	2.14	1	44.98	1.370	(040)	1.371
			{(406)	2.03	1	45.40	1.360	(4.2.10)	1.362
1	28.37	2.04	{(317)	2.03	$\frac{1}{2}$	47.15	1.321	(5.1.11)	1.320

TABLE 4. CHLORITOID AND OTTRELITE: SELECTED ANALYSES  
WITH ATOMIC PROPORTIONS

	1	2	3	4	5	6	7
SiO <sub>2</sub> .....	24.40	24.47	25.30	26.03	24.90	26.19	23.74
Al <sub>2</sub> O <sub>3</sub> .....	42.80	41.34	41.24	42.33	40.36	38.30	39.01
Fe <sub>2</sub> O <sub>3</sub> .....	—	0.38	2.39	4.09	—	6.00	3.04
FeO.....	19.17	18.52	18.02	14.32	26.17	21.11	23.98
MnO.....	—	0.91	0.09	—	—	—	0.49
MgO.....	6.17	6.80	3.60	7.30	2.54	3.30	1.61
CaO.....	—	0.30	—	0.35	—	—	1.02
K <sub>2</sub> O.....	—	—	0.76	—	—	—	—
Na <sub>2</sub> O.....	—	—	2.53	—	—	—	—
TiO <sub>2</sub> .....	—	—	—	—	—	—	—
H <sub>2</sub> O.....	6.90	6.98	6.15	6.56	6.23	5.50	7.72
	99.44	99.70	100.08	100.98	100.20	100.40	100.61
Si.....	1.93	1.94	1.96	1.96	1.98	2.00	2.01
Al.....	2.07	2.06	2.04	2.04	2.02	2.00	1.99
(Si, Al).....	4.00	4.00	4.00	4.00	4.00	4.00	4.00
Al.....	1.91	1.81	1.72	1.71	1.75	1.44	1.90
Fe'''.....	—	0.02	0.14	0.23	—	0.34	0.29
Fe''.....	1.26	1.23	1.17	0.90	1.74	1.35	1.70
Mn.....	—	0.06	0.01	—	—	—	0.04
Mg.....	0.73	0.81	0.41	0.82	0.30	0.38	0.20
Ca.....	—	0.03	—	0.03	—	—	0.09
K.....	—	—	0.08	—	—	—	—
Na.....	—	—	0.38	—	—	—	—
R.....	3.90	3.96	3.91	3.69	3.79	3.51	4.22
OH.....	3.62	3.70	3.17	3.40	3.40	2.80	4.36
O.....	10.00	10.00	10.00	10.00	10.00	10.00	10.00

1. Zermatt, Switzerland; anal. Damour in Des Cloizeaux (1884). 2. Shetland, Scotland; anal. Heddle (1879). 3. Saas Valley, Switzerland; anal. Jacob in Friedlaender (1930). 4. St. Marcel, Italy; anal. Suida, in Tschermak and Sipöcz (1879). 5. Grippe, Ile de Groix, France; anal. Renard, in Barrois (1884). 6. Pregratten, Tyrol; anal. Kobell (1854). 7. Kalgoorlie, W. Australia; anal. Simpson (1931). 8. Crestlianderstobel, Switzerland; anal. Jacob, in Friedlaender (1930). 9. St. Marcel, Italy; anal. Delesse (1846, in Hintze, 1897, p. 677). 10. Pregratten, Tyrol; anal. Sipöcz, in Tschermak and Sipöcz (1879). 11. Champion Mine, Michigan, U. S. A.; anal. Lane and Keller (1891). 12. St. Marcel, Italy; anal. Damour, in Des Cloizeaux (1884). 13. Leeds, Megantic County, Quebec, Canada; anal. Hunt (1861). 14. Krivoy Rog, Ukraine; anal. Tarasenko (1925). 15. Grossarl, Germany; anal. Cathrein (1887). 16. St. Marcel, Italy; anal. Kobell (1853). 17. Hetzschen, Germany; anal. Schröder



TABLE 4—(continued)

	8	9	10	11	12	13	14
SiO <sub>2</sub> .....	26.62	24.10	24.90	24.29	25.50	26.30	27.30
Al <sub>2</sub> O <sub>3</sub> .....	37.65	40.71	40.99	34.00	38.13	37.10	37.80
Fe <sub>2</sub> O <sub>3</sub> .....	11.91	—	0.55	10.55	—	—	6.85
FeO.....	13.91	27.10	24.28	20.52	23.58	25.92	20.82
MnO.....	0.04	—	—	—	—	0.93	—
MgO.....	2.67	—	3.33	1.29	5.19	3.66	0.93
CaO.....	—	—	—	0.59	—	—	—
K <sub>2</sub> O.....	0.46	—	—	0.97	—	—	—
Na <sub>2</sub> O.....	1.21	—	—	0.35	—	—	—
TiO <sub>2</sub> .....	0.21	—	—	0.28	—	—	—
H <sub>2</sub> O.....	5.59	7.24	7.82	6.75	6.90	6.10	6.19
	100.27	99.15	101.87	99.59	99.30	100.01	99.89
Si.....	2.02	2.03	2.04	2.05	2.08	2.09	2.15
Al.....	1.98	1.97	1.96	1.95	1.92	1.91	1.85
(Si, Al).....	4.00	4.00	4.00	4.00	4.00	4.00	4.00
Al.....	1.39	2.07	2.00	1.42	1.74	1.57	1.65
Fe'''.....	0.68	—	0.03	0.67	—	—	0.40
Fe''.....	0.88	1.91	1.66	1.44	1.60	1.72	1.37
Mn.....	—	—	—	—	—	0.06	—
Mg.....	0.30	—	0.41	0.16	0.63	0.44	0.11
Ca.....	—	—	—	0.05	—	—	—
K.....	0.05	—	—	0.12	—	—	—
Na.....	0.18	—	—	0.06	—	—	—
R.....	3.48	3.98	4.10	3.92	3.97	3.79	3.53
OH.....	2.83	4.07	4.27	3.79	3.74	3.23	3.24
O.....	10.00	10.00	10.00	10.00	10.00	10.00	10.00

(1884, in Hintze, 1897, p. 677). 18. Kossoibrod, Ural Mts., Russia; anal. Bonsdorff, in Rose (1837, in Hintze, 1897, p. 677). 19. Dutchess County, N. Y., U. S. A.; anal. Balk and Barth (1934). 20. Deep River Region, N. Carolina, U. S. A.; anal. Stukey (1926).

## COMPOSITION AND CELL CONTENT

The empirical molecular weight of chloritoid is  $M = VG/1.6602 = 1959$ , using the measured specific gravity  $G = 3.528$ . With this value the analysis of chloritoid from Megantic County, Quebec (Hunt, 1861) gives the accompanying empirical cell contents, and assumed grouping of the atoms in equivalent positions in the unit cell.

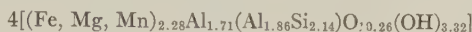
TABLE 4—(continued)

	15	16	17	18	19	20
SiO <sub>2</sub> .....	26.17	25.75	28.04	27.48	29.5	29.28
Al <sub>2</sub> O <sub>3</sub> .....	40.38	37.50	36.19	35.57	38.1	37.98
Fe <sub>2</sub> O <sub>3</sub> .....	—	—	—	—	2.3	2.32
FeO.....	26.41	21.00	29.79	27.05	21.5	21.97
MnO.....	—	—	—	0.30	0.2	0.29
MgO.....	0.08	6.20	1.25	4.29	1.6	1.28
CaO.....	—	—	0.20	—	—	—
K <sub>2</sub> O.....	—	—	—	—	—	—
Na <sub>2</sub> O.....	—	—	—	—	—	—
TiO <sub>2</sub> .....	—	—	—	—	1.2	0.86
H <sub>2</sub> O.....	6.96	7.80	5.88	6.95	5.6	6.04
	100.00	98.25	101.35	101.64	100.0	100.02
Si.....	2.16	2.17	2.20	2.22	2.26	2.27
Al.....	1.84	1.93	1.80	1.78	1.74	1.73
(Si, Al).....	4.00	4.00	4.00	4.00	4.00	4.00
Al.....	2.00	1.80	1.55	1.60	1.69	1.75
Fe'''.....	—	—	—	—	0.13	0.13
Fe''.....	1.78	1.48	1.95	1.82	1.38	1.43
Mn.....	—	—	—	0.02	0.01	0.02
Mg.....	0.01	0.78	0.15	0.52	0.18	0.15
Ca.....	—	—	0.02	—	—	—
K.....	—	—	—	—	—	—
Na.....	—	—	—	—	—	—
R.....	3.79	4.06	3.67	3.96	3.39	3.48
OH.....	3.74	4.39	3.08	3.74	2.85	3.13
O.....	10.00	10.00	10.00	10.00	10.00	10.00

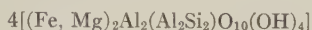
SiO <sub>2</sub> .....	26.30	Si.....	8.58	16.00
Al <sub>2</sub> O <sub>3</sub> .....	37.10	Al.....	14.26	
FeO.....	25.92	Fe.....	7.07	15.95
MnO.....	0.93	Mn.....	0.26	
MgO.....	3.66	Mg.....	1.78	
H <sub>2</sub> O.....	6.10	OH.....	13.26	
		O.....	41.03	
	100.01			

The similarity in geometrical properties between chloritoid and the micas indicates a sheet structure with the characteristic (Si,Al)<sub>4</sub>O<sub>10</sub> ratio. On this basis part of the Al has as usual been grouped with Si to make 16

atoms (oxygen=40) and the remainder placed with the other cations. The ratio of Al to Si is approximately 2:2 in chloritoid in contrast with 1:3 in the micas. The cell content may be written:

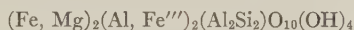


This differs from the probable ideal cell content:

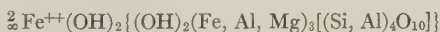


mainly in the term (OH) which may be low, due to analytical error.

The better available analyses of chloritoid are presented in Table 4, with atomic proportions calculated to oxygen, exclusive of hydroxyl, equals 10. Sufficient Al has been grouped with Si to make (Si, Al)=4, and it is clear that the ratio Si:Al in this group is consistently near 2:2. The remaining Al has been grouped with Fe''', Fe'', Mn, Mg, Ca, K, Na, to give total R adding to nearly 4. In this group Al or (Al, Fe''') amounts to nearly 2 and the rest is essentially (Fe'', Mg). Mn, Ca, K, Na are apparently unessential and occasional TiO<sub>2</sub> is neglected since rutile inclusions have been reported in chloritoid. (OH) ranges from about 3 to 4, with 4 evidently the full number. The composition of chloritoid is thus



in agreement with the formula derived from the studied material. This formula compares with the early empirical formula, H<sub>2</sub>(Fe, Mg)Al<sub>2</sub>SiO<sub>7</sub>; and in showing the (Si, Al)<sub>4</sub>O<sub>10</sub> groups of the platy silicates it has something in common with a structural formula suggested for chloritoid by Machatschki and Mussung (1942):



The present paper prepares the way for a determination of the structure of chloritoid but this has not been attempted.

In conclusion, the author wishes to acknowledge the guidance and helpful supervision of Dr. M. A. Peacock in the course of this study and in the preparation of this paper for publication.

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# STUDIES OF NATURAL AND ARTIFICIAL SELENIDES: I—KLOCKMANNITE, $\text{CuSe}^1$

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## ABSTRACT

$\text{CuSe}$  prepared by dry fusion and by hydrosynthesis is physically and structurally identical with klockmannite from Sierra de Umango, Argentina. Pyrosynthetic  $\text{CuSe}$  gave  $G=5.99$ . Minute greenish black hexagonal plates of hydrosynthetic  $\text{CuSe}$  show  $c(0001)$ ,  $n(10\bar{1}2)$ ,  $r(10\bar{1}1)$ ,  $m(10\bar{1}0)$ ; the unit cell with space group  $C6/mmc$ ,  $a=3.93$ ,  $c=17.22$  kX, contains  $6[\text{CuSe}]$ . A superstructure has  $a'=12a$ ,  $c'=c$ . Klockmannite is clearly isostructural with covellite.

Klockmannite (Ramdohr, 1928) is a rare copper selenide which occurs intimately associated with umangite, clausthalite, eucairite, and chalcomenite at Sierra Umango (Argentina), and sparingly with other selenides at Lerbach and Tilkerode (Harz Mts.) and Skrikerum (Sweden). Unlike umangite (Klockmann, 1891), which is reddish violet on fresh surfaces, klockmannite is slate-grey; but since both minerals finally tarnish to a dull bluish black color they were not distinguished by Klockmann and the slate-grey mineral was actually first recognized and described by Ramdohr on a specimen which had been labelled "umangite" by Klockmann. Analyses of somewhat impure materials have indicated the compositions  $\text{Cu}_3\text{Se}_2$  for umangite and  $\text{CuSe}$  for klockmannite. Ramdohr (1928, 1931) has added a wealth of microscopic observations on both minerals and also brief crystallographic observations on crystals said to be artificial  $\text{CuSe}$ . These data all tend to establish klockmannite as a distinct species,  $\text{CuSe}$ , analogous to covellite,  $\text{CuS}$ ; but the evidence for the composition of the selenide is hardly adequate and further work on natural and artificial material are needed to confirm the individuality and properties of the mineral.

Only one authentic specimen of klockmannite could be obtained, Royal Ontario Museum, M 17025, from Sierra de Umango, Argentina. This specimen had been supplied by Professor Paul Ramdohr in an exchange of minerals and it was kindly lent for study by Dr. V. B. Meen. On this specimen the somewhat scanty klockmannite is tarnished and not very different from umangite in appearance, and it is intergrown by much chalcomenite. For lack of material further observations on the mineral were limited to obtaining the  $x$ -ray powder pattern, which proved to

<sup>1</sup> Extracted from an unpublished M.A. thesis: Studies of copper selenide minerals—Queen's University (Kingston, Ontario), 1947. In this work the helpful supervision of Professors J. E. Hawley and L. G. Berry is gratefully acknowledged.

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be quite unlike that of umangite. The remaining observations were made on artificial CuSe.

*Preparation of CuSe.* Pyrosynthetic CuSe was prepared by fusing copper and selenium metals in equal atomic proportions in evacuated silica glass tubes at 1125° C. Two fusions were prepared in this way; one was quenched in cold water from 1125° C., the other was cooled slowly to room temperature. In polished sections the products are homogeneous, and they yielded *x*-ray powder patterns identical with the pattern given by the available specimen of klockmannite. The pattern of the material which was cooled slowly is sharper, due probably to better crystalline development.

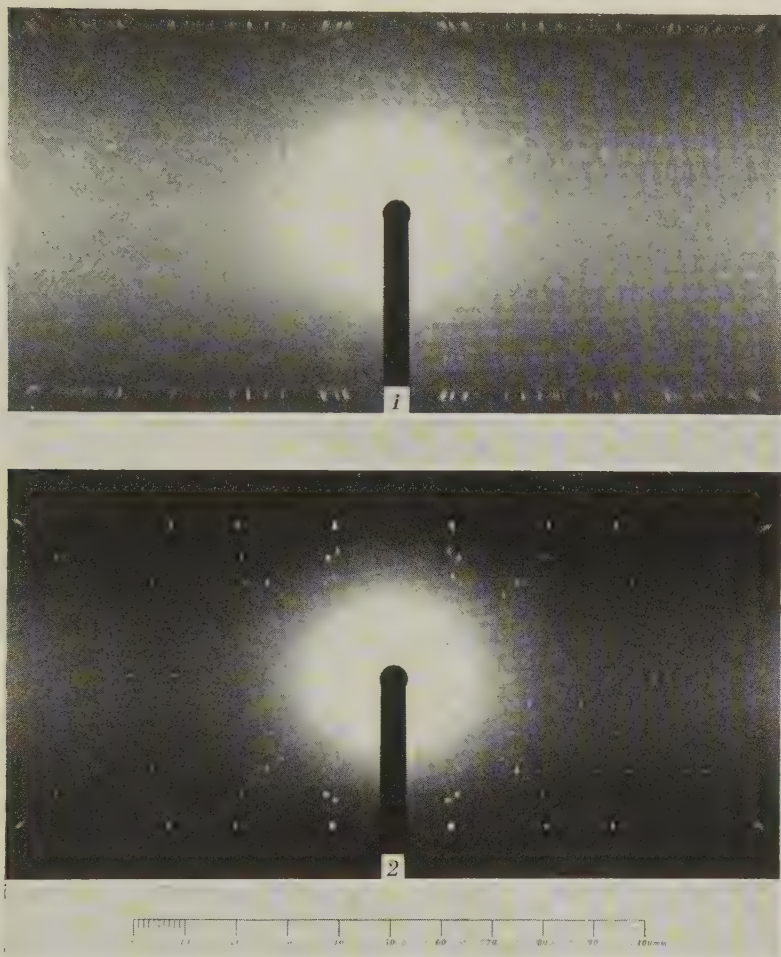
Crystals of artificial klockmannite were also produced by hydrosynthesis from a 0.5 molar solution of copper sulphate in contact with copper and selenium metal in a steel bomb at about 400° C. The bomb is of the same design as that used by Smith (1947) for the hydrosynthesis of vein minerals. The synthetic crystals are greenish black plates showing hexagonal outline, seldom more than 0.1 mm. wide. The *x*-ray powder pattern of these crystals is also identical with the pattern of klockmannite.

*Physical Properties.* Six determinations of specific gravity, made with the Berman balance on fragments of the pyrosynthetic material gave an average value 5.99. The mineral specimen did not yield fragments of sufficient size or purity for specific gravity determinations, but Ramdohr (1928) gives the specific gravity as >5. In the natural specimen, klockmannite is finely granular and cut by veinlets of chalcomenite. In reflected light a polished section of klockmannite shows strong reflection pleochroism varying from bluish green to white. It is strongly anisotropic with four extinction positions and polarization colors from brownish blue to fiery orange, giving a mottled appearance to the section. The Talmadge hardness is C, estimated with a needle. Etch tests: HNO<sub>3</sub> fumes tarnish; HCl negative; KCN stains black rapidly; FeCl<sub>3</sub> negative, KOH stains differentially brown; HgCl<sub>2</sub> stains differentially blue. These reactions agree with those reported by Short (1940, p. 136).

*Structural Crystallography.* Cleavage fragments suitable for single-crystal measurements were not obtainable from the natural specimen or the artificial material made by dry fusion. A hydrosynthetic crystal plate (0.1 mm. in width) was adjusted to rotate about an edge of the hexagonal plate, an *a*-axis, for *x*-ray measurements. Later the crystal was readjusted to rotate about the normal to the plate, the *c*-axis. Good rotation and Weissenberg photographs were obtained with CuK radiation. Measurements on the films gave the hexagonal cell dimensions:<sup>3</sup>

$$a = 3.94 \pm 0.01 \text{ kX}, c = 17.26 \pm 0.05 \text{ kX}; c/a = 4.38$$

<sup>3</sup> Using  $\lambda \text{ CuK}\alpha_1 = 1.5374 \text{ kX}$ .



FIGS. 1, 2. Artificial CuSe: X-ray rotation photographs with CuK radiation (long exposure); camera radius  $90/\pi$  mm. FIG. 1. Rotation about  $a$ -axis, showing weak intermediate layer lines requiring a period of  $12a$ . FIG. 2. Rotation about  $c$ -axis.

A heavily exposed photograph about the  $a$ -axis (Fig. 1) shows very weak intermediate layer lines which indicate a 12-fold multiplicity of the  $a$ -length. The rotation about the  $c$ -axis (Fig. 2) shows a few weak lines of nearly continuous diffraction parallel to the Bernal curves.

The Laue symmetry is  $6/mmm$  and the systematically missing spectra are  $(h \cdot h \cdot 2\bar{h} \cdot l)$  present only with  $l = 2n$ . These conditions are characteristic of the space-group  $D_{6h}^{4}-C6/mmc$ . An additional condition,  $(h\bar{k}i5)$  all absent, is due to a structural peculiarity.

*Geometrical Crystallography.* Klockmannite occurs in granular aggre-

gates and no recognizable crystals are developed. Cleavage is not visible in our hand specimen of the natural material, although reported by Ramdohr as (0001), perfect. The material made by dry fusion shows one perfect cleavage but the hydrosynthetic crystals are too small to permit the observation of cleavage. On the reflecting goniometer such crystals show platy development of  $c(0001)$  and six-sided outlines due to the intersections of this plane with  $n(10\bar{1}2)$ ,  $r(10\bar{1}1)$ , and  $m(10\bar{1}0)$ . The edge-faces are very narrow but give fair reflections. The following measured angles were obtained from two crystals:

$$\begin{array}{l} n(10\bar{1}2) \quad \rho = 67^\circ 51' - 68^\circ 30' \quad (9) \quad \text{av. } 68^\circ 13' \\ r(10\bar{1}1) \quad \rho = 77^\circ 38' - 79^\circ 23' \quad (8) \quad \text{av. } 78^\circ 33' \end{array}$$

These angles yield the axial ratio  $c/a = 4.31$  which agrees roughly with the structural lattice ratio,  $c/a = 4.38$ , the axial ratio,  $c/a = 4.382$ , given by Ramdohr (1931, p. 317) for artificial crystals. Thus the  $x$ -ray measurements confirm the symmetry and setting chosen by Ramdohr.

*Composition and Cell Content.* The cell dimensions and measured specific gravity on artificial CuSe, with the mass factor 1.650, give the molecular weight of the cell contents  $M = 836.2$ . This molecular weight

TABLE 1. KLOCKMANNITE FROM SIERRA DE UMANGO, ARGENTINA  
ANALYSIS AND CELL CONTENT

1		2		3		4	
Cu	35.37	Cu	24.14	Cu	43.58	Cu	5.73
Se	45.73	Se	31.67	Se	56.42	Se	5.98
Pb	0.84	CuSeO <sub>3</sub> · 2H <sub>2</sub> O	40.25				
Ag	0.73	Pb	0.84				
Fe <sub>2</sub> O <sub>3</sub>	0.74	Ag	0.73				
Quartz	1.34	Fe <sub>2</sub> O <sub>3</sub>	0.74				
H <sub>2</sub> O+	6.84	SiO <sub>2</sub>	1.34				
H <sub>2</sub> O—	0.48	H <sub>2</sub> O—	0.48				
[92.07]		100.19		100.00			

1. Anal. Geilmann, in Ramdohr (1928). 2. Anal. 1, as recast in Ramdohr (1928), showing chalcomenite (CuSeO<sub>3</sub> · 2H<sub>2</sub>O) determined by solution in ammonia. 3. Recast analysis recalculated to 100 per cent after deducting CuSeO<sub>2</sub> · 2H<sub>2</sub>O, PbSe, Ag<sub>2</sub>Se, Fe<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>. 4. Empirical cell content.

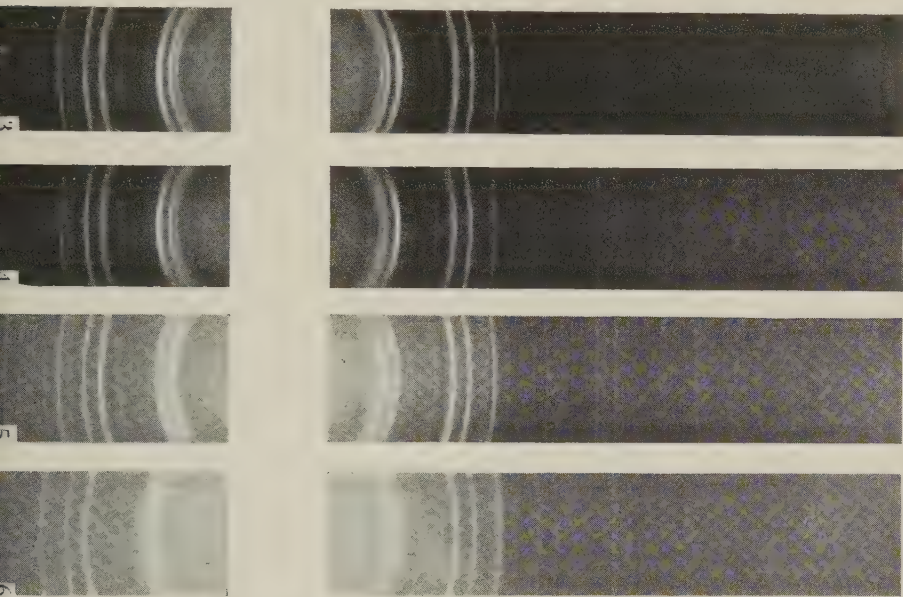
combined with the analysis given by Ramdohr (1928) (Table 1), recalculated to 100 per cent after deducting chalcomenite (CuSeO<sub>3</sub> · 2H<sub>2</sub>O), clausthalite (PbSe), naumannite (Ag<sub>2</sub>Se), Fe<sub>2</sub>O<sub>3</sub>, and SiO<sub>2</sub>, indicates the structural cell content Cu<sub>6</sub>Se<sub>6</sub>.

The empirical formula CuSe is confirmed by the fact that homogeneous artificial material identical with klockmannite was formed by fusion of



the elements in equal atomic proportions in a closed system. The specific gravity calculated for the unit cell containing  $6[\text{CuSe}]$  is 6.12, in fair agreement with the measured value 5.99.

*X-Ray Powder Photographs.* Identical patterns are given by natural klockmannite (Fig. 3), pyrosynthetic CuSe (Fig. 4), and hydrosynthetic crystals of CuSe (Fig. 5). Table 2 gives the observed relative intensities



FIGS. 3-6. X-ray powder photographs with Cu/Ni radiation; camera radius  $90/\pi$  mm. ( $1^\circ = 1$  mm. on film); full size reproductions of contact prints. FIG. 3. Klockmannite Sierra de Umango, Argentina. FIG. 4. Artificial CuSe formed by pyrosynthesis. FIG. 5. Artificial CuSe crystals formed by hydrosynthesis. FIG. 6. Covellite, Butte, Montana.

and glancing angles for  $\text{CuK}\alpha$  radiation, together with the measured spacings, the indices of the powder lines, and the calculated spacings, using the cell dimensions,  $a = 3.93$ ,  $c = 17.22$  kX, which are slightly smaller and more accurate than the values obtained from the single-crystal measurements. The measured spacings given by Harcourt (1942) for klockmannite from Sierra de Umango agree fairly well with the stronger lines of our pattern.

The close similarity in lattice dimensions, crystal form and structural formula between klockmannite and covellite confirms the already inferred isostructural character of the two minerals. The close structural similarity is further borne out by comparing the powder patterns of the two minerals (Figs. 3-6).

TABLE 2. KLOCKMANNITE—CuSe: X-RAY POWDER PATTERN

Hexagonal,  $C6/mmc$ ;  $a=3.93$ ,  $c=17.22$  kX,  $Z=6$ 

<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	( <i>hkl</i> )	<i>d</i> (calc.)	<i>I</i>	$\theta(\text{Cu})$	<i>d</i> (meas.)	( <i>hkl</i> )	<i>d</i> (calc.)
6	13.33	3.34	(10 $\bar{1}$ 1)	3.339	$\frac{1}{2}$	39.59	1.206	(2.0. $\bar{2}$ .10)	1.210
9	14.03	3.17	(10 $\bar{1}$ 2)	3.165	$\frac{1}{2}$	40.89	1.174	(2 $\bar{1}$ $\bar{3}$ 6)	1.174
1	15.23	2.93	(10 $\bar{1}$ 3)	2.928	4	41.69	1.156	(1.0. $\bar{1}$ .14)	1.157
10	15.53	2.87	(0006)	2.870	2	42.59	1.136	(30 $\bar{3}$ 0)	1.135
3	20.54	2.19	(10 $\bar{1}$ 6)	2.194	3	44.20	1.103	(2 $\bar{1}$ $\bar{3}$ 8)	1.104
$\frac{1}{2}$	20.95	2.15	(0008)	2.152	$\frac{1}{2}$	45.20	1.083	(1.0. $\bar{1}$ .15)	1.088
4	22.65	1.996	(10 $\bar{1}$ 7)	1.994	$\frac{1}{2}$	45.60	1.076	(0.0.0.16)	1.076
8	23.05	1.963	(11 $\bar{2}$ 0)	1.965	1	46.90	1.053	(30 $\bar{3}$ 6)	1.055
$\frac{1}{2}$	23.75	1.909	(11 $\bar{2}$ 2)	1.916	3	50.41	0.997	(2.0. $\bar{2}$ .14)	0.997
6	25.05	1.815	(10 $\bar{1}$ 8)	1.819	1	51.51	0.982	(2 $\bar{2}$ 40)	0.982
$\frac{1}{2}$	25.56	1.782	(11 $\bar{2}$ 4)	1.788	$\frac{1}{2}$	51.92	0.977	(2 $\bar{2}$ 42)	0.976
1	26.96	1.695	{(20 $\bar{2}$ 0)	1.702	$\frac{1}{2}$			(30 $\bar{3}$ 9)	0.976
			{(20 $\bar{2}$ 1)	1.694				{(1.1. $\bar{2}$ .16)	0.944
			{(20 $\bar{2}$ 2)	1.669				{(31 $\bar{4}$ 0)	0.944
1	27.36	1.673	{(10 $\bar{1}$ 9)	1.668	3	54.52	0.944	{(31 $\bar{4}$ 1)	0.943
			{(11 $\bar{2}$ 6)	1.621	$\frac{1}{2}$	55.72	0.930	{(2 $\bar{2}$ 46)	0.930
5	28.36	1.619	(11 $\bar{2}$ 6)	1.621	$\frac{1}{2}$	59.03	0.896	{(31 $\bar{4}$ 6)	0.897
$\frac{1}{2}$	30.07	1.534	(1.0. $\bar{1}$ .10)	1.536	$\frac{1}{2}$	59.83	9.889	{(3.0. $\bar{3}$ .12)	0.890
$\frac{1}{2}$	31.57	1.468	(20 $\bar{2}$ 6)	1.464	4			{(2.1. $\bar{3}$ .14)	0.889
$\frac{1}{2}$	32.37	1.436	(0.0.0.12)	1.435	2	62.74	0.865	{(31 $\bar{4}$ 8)	0.865
$\frac{1}{2}$	32.77	1.420	(1.0. $\bar{1}$ .11)	1.422	$\frac{1}{2}$	63.74	0.857	{(2.1. $\bar{3}$ .15)	0.857
1	33.37	1.398	(20 $\bar{2}$ 7)	1.399				{(1.0. $\bar{1}$ .20)	0.834
3	35.28	1.331	(20 $\bar{2}$ 8)	1.335	1	67.15	0.834	{(3.0. $\bar{3}$ .14)	0.834
$\frac{1}{2}$	36.78	1.284	{(21 $\bar{3}$ 0)	1.286				{(2.0. $\bar{2}$ .18)	0.834
			{(21 $\bar{3}$ 1)	1.283	$\frac{1}{2}$	68.55	0.826	{(2.1. $\bar{3}$ .16)	0.826
1	37.08	1.275	{(21 $\bar{3}$ 2)	1.272	$\frac{1}{2}$	74.66	0.797	{(1.0. $\bar{1}$ .21)	0.797
			{(20 $\bar{2}$ 9)	1.272				{(2.1. $\bar{3}$ .17)	0.796
$\frac{1}{2}$	37.78	1.255	(21 $\bar{3}$ 3)	1.255	2	79.27	0.782	(0.0.0.22)	0.783
			{(1.0. $\bar{1}$ .13)	1.234					
$\frac{1}{2}$	38.58	1.233	(21 $\bar{3}$ 4)	1.232					
			(0.0.0.14)	1.230					

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# CRYSTAL STRUCTURE OF RICKARDITE, $\text{Cu}_{4-x}\text{Te}_2$ <sup>1</sup>

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## ABSTRACT

Rickardite and the identical artificial compound  $\text{Cu}_{4-x}\text{Te}_2$  have the C38-type ( $\text{Cu}_2\text{Sb}$ ) of structure. Tetragonal,  $P4/nmm$ ;  $a = 3.97$ ,  $c = 6.11$  kX; 2Cu in ( $a$ ): 000,  $\frac{1}{2}\frac{1}{2}0$ ;  $(2-x)\text{Cu}$  in ( $c$ ):  $0\frac{1}{2}z_1$ ,  $\frac{1}{2}0\bar{z}_1$  with  $z_1 = 0.27 \pm 0.01$ ; 2Te in ( $c$ ):  $0\frac{1}{2}z_2$ ,  $\frac{1}{2}0\bar{z}_2$ , with  $z_2 = 0.715 \pm 0.005$ . The structure is grossly defective, with  $x$  nearly constant and equal to 1.2 or roughly 1. The formula  $\text{Cu}_4\text{Te}_3$  hitherto given for rickardite and for the artificial compound should therefore be replaced by the cell formula or, for simplicity, by  $\text{Cu}_3\text{Te}_2$ . The variously blue colours of rickardite and other copper ores containing  $\text{Cu}^{\text{II}}$  is attributed to Cu atoms in the higher than normal valence state.

Rickardite<sup>4</sup> is a natural copper telluride which was discovered in the ore of the Good Hope Mine, Vulcan, Gunnison County, Colorado, by Dr. Loui Weiss, and was subsequently named and described by Ford (1903). The mineral is massive and brittle, with hardness  $3\frac{1}{2}$  and specific gravity 7.54. It has metallic lustre and a remarkable purple-red colour on freshly fractured surfaces. Duplicate analyses indicated the composition  $\text{Cu}_4\text{Te}_3$ . At Vulcan, rickardite is associated with tellurium, petzite, berthierite, and pyrite. The mineral has also been reported at the Empress Josephine mine, Bonanza, Colorado (Patton, 1916, p. 108), the San Sebastian mine, Salvador (Wuensch, 1917, p. 348), the Junction mine, Warren, Arizona (Crawford, 1930, p. 272), the Kalgurli mine, Kalgoorlie, Western Australia (Stillwell, 1931, p. 186), and the Horne mine, Noranda, Quebec (Price, 1934, p. 132); but these reports contain no significant addition to the meagre description of the mineral.

Polished sections of rickardite have been described by Ramdohr (1937, p. 204) and Short (1940, p. 113). Ramdohr remarks that the formula  $\text{Cu}_4\text{Te}_3$  is not certain and that the composition might be  $\text{Cu}_3\text{Te}_2$ , by analogy with the mineral umangite,  $\text{Cu}_3\text{Se}_2$ , which, however, gives a quite dissimilar x-ray powder pattern. The reflecting power is moderate, the reflection pleochroism is very strong and striking (carmine-red to violet-grey), and the anisotropism is enormous (canary-yellow to deep brown-red). Some sections are optically isotropic, indicating that rickardite is hexagonal or tetragonal. The curious porous texture of Ramdohr's section and the arrangement of the individual grains indicated that the

<sup>1</sup> Based on an unpublished M.A. thesis by S. A. Forman: The crystal structure of rickardite,  $\text{Cu}_{2-x}\text{Te}$ —*University of Toronto*, 1946.

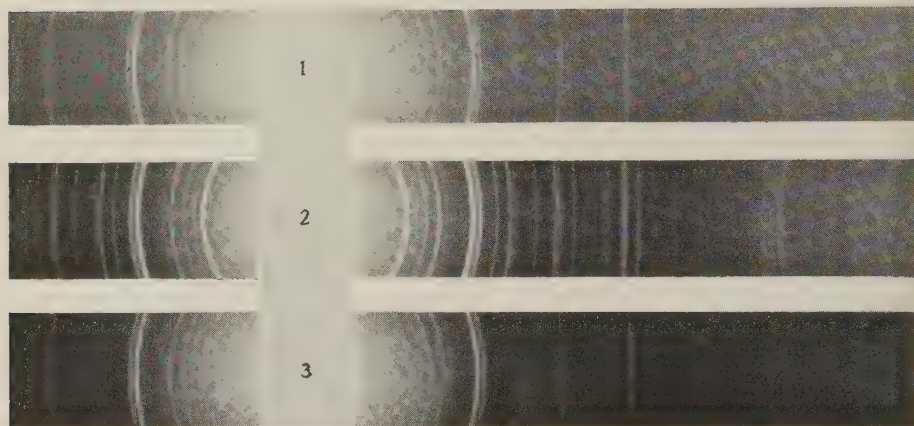
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<sup>3</sup> Professor of Crystallography and Mineralogy, Department of Geological Sciences.

<sup>4</sup> Rickardite and weissite are briefly described by Thompson (1949).

whole mass originated as a pseudomorph of a probably cubic mineral. Short also remarks on the similarity of rickardite and umangite, and he describes four extinctions per revolution between crossed nicols with fiery orange interference colours. X-ray information on rickardite is limited to powder spacings and intensities by Waldo (1935, p. 587) and Harcourt (1942, p. 96). Thus rickardite is a mineral of striking and unmistakable appearance; at the same time the description of the mineral is unsatisfactory since it appears to be an inversion product of somewhat uncertain composition and almost unknown crystallography.

Intimately associated with rickardite is a telluride of copper which differs from rickardite in composition and colour. This mineral was distinguished from rickardite by Crawford (1927) who first described it



FIGS. 1-3. X-ray powder photographs with Cu/Ni radiation; camera radius,  $360/4\pi$  mm.; actual size reproductions of contact prints. Fig. 1. Rickardite. Fig. 2. Alloy with Cu:Te=4:3. Fig. 3. Alloy with Cu:Sb=4:3.

under the name weissite. This second copper telluride is likewise massive, with hardness 3, and specific gravity about 6; it has metallic lustre, a bluish black colour tarnishing deep black, and a composition given by an unusual type of formula  $\text{Cu}_5\text{Te}_3$ . Ramdohr (1938) and Short (1940, p. 113) give observations on polished sections, which are light grey like ordinary chalcocite in reflected light, with practically no reflection pleochroism but distinct anisotropism of orthorhombic character. From the similarity to chalcocite Short suggests that the proper composition of weissite is  $\text{Cu}_2\text{Te}$ . Thus weissite seems to be even less well defined than rickardite and it is not surprising that L. J. S. [pencer] (*Min. Abs.* 3, 368) considered weissite to be the same as rickardite.



The system Cu-Te was studied in detail by Chikashige (1907, in Hansen, 1936, p. 647) without reference to the natural compounds. Two compounds were recognized:  $\text{Cu}_2\text{Te}$ , which takes up to 5% Te in solid solution and apparently passes through two transformations, at  $387^\circ$  and  $360^\circ$ ; and  $\text{Cu}_4\text{Te}_3$ , which inverts at  $365^\circ$  to a low temperature form. Watanabe (in Ramdohr, 1938) notes that weissite corresponds to artificial  $\text{Cu}_2\text{Te}$  with 5% added Te; rickardite presumably corresponds to the artificial compound  $\text{Cu}_4\text{Te}_3$ . The results on artificial tellurides appear to support the individuality of both rickardite and weissite. The unusual

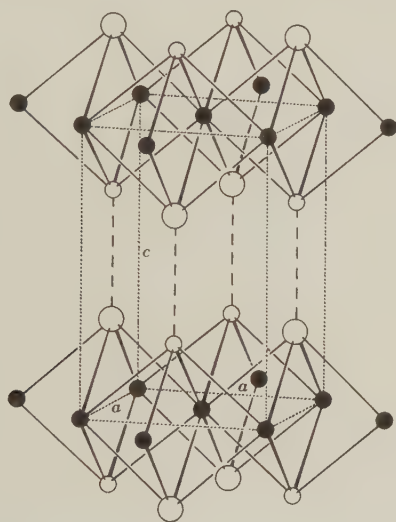


FIG. 4. Rickardite structure; small filled circles are fully occupied Cu positions; small blank circles are partially (roughly half) filled Cu positions; larger blank circles are Te positions.

composition of the former,  $\text{Cu}_4\text{Te}_3$ , seems to be confirmed, while the latter appears to have a defective  $\text{Cu}_2\text{Te}$  composition rather than the definite composition  $\text{Cu}_5\text{Te}_3$ .

The present work is concerned only with rickardite, the purple-red mineral with empirical composition  $\text{Cu}_4\text{Te}_3$ . To verify the probable identity of this mineral with the compound  $\text{Cu}_4\text{Te}_3$  reported in the system Cu-Te, a charge of the powdered elements in the proportions Cu:Te = 4:3 was fused in an evacuated silica glass tube and cooled in air. The product was a nearly homogeneous platy crystalline product with the characteristic purple-red colour of rickardite and a distinct pinakoidal cleavage parallel to the plates. This product gave an x-ray powder pattern (Fig. 2) which is identical with that of the mineral, (Fig. 1), con-

firming the presumed identity of rickardite and the artificial compound with the empirical composition  $\text{Cu}_4\text{Te}_3$ . The phase diagram in Hansen (1936, p. 648) indicates that this compound does not vary considerably in composition.

Ramdohr's recognition of the uniaxial character of rickardite proved to be well founded. A study of the spacings of the fairly simple powder pattern led to a tetragonal lattice with relatively small dimensions:<sup>5</sup>

$$a=3.97, c=6.11 \text{ kX}$$

On this lattice the powder pattern was completely indexed with good agreement between measured and calculated spacings, as shown later in Table 1. The systematically missing spectra lead to the single condition,  $(hk0)$  present only with  $(h+k)$  even. This condition is characteristic of the space-groups  $P4/n$  and  $P4/nmm$ , in both of which there are no odd numbers of equivalent positions. From the mean values of the duplicate analyses of rickardite, Cu 40.74, Te 59.21 (Ford, 1903), the measured specific gravity 7.54, the cell dimensions given above, and the mass factor 1.650, the computed cell content is Cu 2.82, Te 2.04 atoms. Clearly the structural formula cannot be  $\text{Cu}_4\text{Te}_3$ , which appeared to be so well established. The ideal cell content must be  $\text{Cu}_4\text{Te}_2$  and therefore the structural formula must be written  $\text{Cu}_{4-x}\text{Te}_2$  indicating a deficiency in Cu amounting in this case to roughly one atom per unit cell.

Before attempting to determine the atomic arrangement of the copper telluride, the *Strukturbericht* was searched for analogous structures. It was soon found that the structure of the artificial compound  $\text{Cu}_2\text{Sb}$  (C38-type, SB, 1937, 33), determined by Elander, Hägg & Westgren (1935), would probably prove to be the arrangement of the copper telluride.  $\text{Cu}_2\text{Sb}$  is tetragonal with the space-group  $P4/nmm$ , which is one of the two space-groups allowed for  $\text{Cu}_{4-x}\text{Te}_2$ ; the cell dimensions of  $\text{Cu}_2\text{Sb}$  are:

$$a=3.992, c=6.091$$

which are very similar to those of rickardite; the powder intensities of the two compounds are nearly alike, and the antimonide even has a noteworthy violet colour to compare with the purple-red of the telluride.

In the structure of  $\text{Cu}_2\text{Sb}$  the atoms are placed as follows:

$$\begin{aligned} 2 \text{ Cu in } (a): & 000; \frac{1}{2}\frac{1}{2}0 \\ 2 \text{ Cu in } (c): & 0\frac{1}{2}z_1; \frac{1}{2}0\bar{z}_1, \text{ with } z_1=0.27 \\ 2 \text{ Sb in } (c): & 0\frac{1}{2}z_2; \frac{1}{2}0\bar{z}_2, \text{ with } z_2=0.70 \end{aligned}$$

The structural problem of rickardite thus reduces to finding the distribution of approximately three Cu atoms over the four Cu positions and determining the special values of the two parameters,  $z_1, z_2$ .

<sup>5</sup> Using  $\lambda \text{ CuK}\alpha_1=1.5374 \text{ kX}$ .

The three Cu atoms may be distributed over the four Cu positions in the structure of rickardite in only three possible ways which would not degrade the symmetry of the structure:

1) Three Cu atoms distributed statistically over the four positions so that in effect we would have scattering due to  $\frac{3}{4}$ Cu at each of the four positions.

2) One Cu atom at each of the two positions in (a) and the remaining Cu atom distributed over the two positions in (c), giving the effect of  $\frac{1}{2}$ Cu atom in each of the (c) positions.

3) One Cu atom in each of the (c) positions and the remaining Cu atom distributed over the two positions in (a), giving the effect of  $\frac{1}{2}$ Cu atom in each of the (a) positions.

A choice between these three possibilities was made by comparing measured and calculated intensities<sup>6</sup> for (*h**k*0) reflections which are unaffected by the variable parameters. The results of this comparison are shown below; it is clear that only the second possibility, namely two Cu atoms in the (a) positions and one Cu atom distributed over the (c) posi-

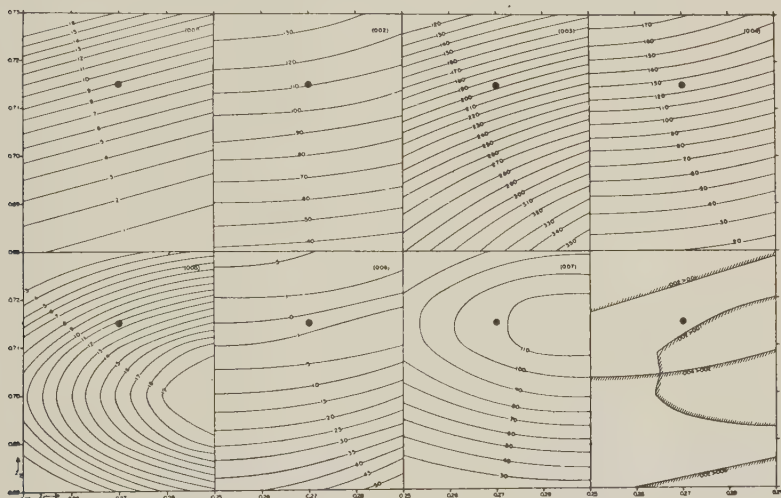


FIG. 5. Calculated intensities of (00*l*) planes with variation of  $z_1$ ,  $z_2$ , to compare with relative Weissenberg intensities.

tions gives calculated values which correspond to those observed on the natural and artificial material.

	(110)	(200)	(220)	(130)
1) 3 Cu in (a) and (c):	177	197.5	59	27.7
2) 2 Cu in (a), Cu in (c):	95	197.5	59	15.2
3) Cu in (a), 2 Cu in (c):	285	197.5	59	43.9
Observed { Rickardite:	2	4	2	0
Artificial Compound:	3	5	2	0

<sup>6</sup> Using  $I \propto NLP|F_0|^2$  with  $f_0$  values from *ITDCS* (1935).

For the purpose of determining the two variable parameters,  $z_1$  which applies to the two  $\frac{1}{2}\text{Cu}$  atoms in (c) and  $z_2$ , the two Te atoms in (c), the powder intensities on the natural and artificial materials were supplemented by intensities of (00*l*) reflections given by a minute basal cleavage plate which had been rotated about a random axis in the basal plane. Packing considerations indicated that the parameters to be determined would certainly lie within the ranges  $z_1=0.25$  to  $0.29$ ,  $z_2=0.68$  to  $0.73$ , and therefore these limits were chosen for the computations represented in the contour maps showing the variation of intensity with both parameters for planes of the type (00*l*) (Fig. 5). In the last square of this figure, the areas excluded by the observed relative intensities of the (00*l*) reflections from the crystal plate are fenced off, leaving a permissible region which restricts  $z_2$  fairly definitely but imposes little restriction on  $z_1$ .

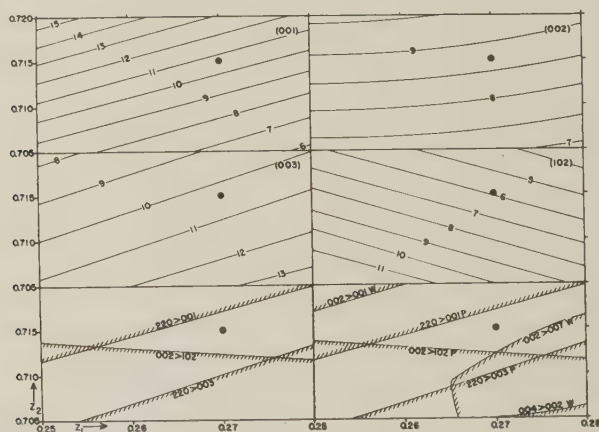


FIG. 6. Calculated intensities of selected planes with variation of  $z_1$ ,  $z_2$ , to compare with certain relative Weissenberg and powder intensities.

In an endeavour to restrict both parameters a little more sharply, a second set of intensity maps was prepared to compare with the powder intensities for planes (00*l*), (002), (003), (102) (Fig. 6). Below these maps the permissible area has been delimited, on the left by certain observed relative powder intensities (*P*) and on the right by two additional single crystal intensities (*W*). In this way  $z_2$  is fixed fairly sharply at  $0.715 \pm 0.005$ ;  $z_1$  is less sharply restricted, the best value being  $0.27 \pm 0.01$ . The relatively wide permissible range of  $z_1$  is due to the fact that this is the parameter of the single Cu atom distributed over the (c) positions; this atom has only about one-quarter of the scattering power of the two Te atoms fixed by  $z_2$ .

These values of  $z_1$  and  $z_2$  give the following comparison of calculated



TABLE 1. RICKARDITE AND ARTIFICIAL  $\text{Cu}_{4-x}\text{Te}_2$ : OBSERVED AND CALCULATED SPACINGS AND POWDER INTENSITIESTetragonal,  $P4/nmm$ ;  $a = 3.97$ ,  $c = 6.11$  kX

$d(\text{meas.})^7$	$d(\text{calc.})$	$(hkl)$	$I(\text{calc.})^8$	$I(\text{obs.})$	
				Rickardite <sup>7</sup>	Artif. <sup>8</sup>
6.05	6.110	(001)	1.29	$\frac{1}{2}$	2
3.35	3.329	(011)	6.56	6	8
3.03	3.055	(002)	1.07	1	3
2.81	2.807	(110)	2.28	2	3
2.54	2.551	(111)	3.90	4	4
2.42	2.421	(012)	0.73	$\frac{1}{2}$	2
2.07	2.067	(112)	10.00	10	10
2.05	2.037	(003)	1.28	1	2
1.984	1.985	(020)	4.80	4	5
—	1.888	(021)	0.18	—	—
1.816	1.812	(013)	0.71	1	2
1.703	1.705	(121)	1.98	2	3
1.665	1.665	(022)	0.71	$\frac{1}{2}$	1
—	1.649	(113)	0.07	—	—
1.528	1.535	(122)	0.45	1	2
	1.528	(004)	0.60		
1.421	1.426	(014)	0.41	2	3
	1.422	(023)	1.39		
1.404	1.404	(220)	1.43	2	2
—	1.368	(221)	0.06	—	—
1.339	1.342	(114)	0.10	1	2
	1.338	(123)	0.48		
1.292	1.293	(031)	0.37	$\frac{1}{2}$	$\frac{1}{4}$
1.276	1.275	(222)	0.28	—	$\frac{1}{4}$
1.254	1.255	(130)	0.37	$\frac{1}{2}$	$\frac{1}{4}$
1.229	1.230	(131)	0.70	$\frac{1}{2}$	$\frac{1}{2}$
—	1.222	(005)	0.04	—	—
—	1.214	(032)	0.08	—	—
1.209	1.211	(024)	1.09	$\frac{1}{2}$	3
—	1.168	(015)	0.03	—	—
1.156	1.161	(132)	2.65	3	5
	1.158	(124)	0.41		
	1.156	(223)	0.70		
1.121	1.120	(115)	1.11	1	2
—	1.110	(033)	0.14	—	—
1.083	1.084	(231)	0.47	1	1
—	1.069	(133)	0.04	—	—
—	1.041	(025)	0.12	—	—
1.034	1.036	(232)	0.11	$\frac{1}{2}$	$\frac{1}{2}$
	1.034	(224)	0.72		
—	1.018	(006)	0.00	—	—
0.986	0.986	(016)	0.25	—	$\frac{1}{2}$

<sup>7</sup> By R. M. Thompson; routine observations for a standard determinative powder pattern.<sup>8</sup> By S.A.F.

and observed intensities for single crystal (00*l*) reflections:

	(001)	(002)	(003)	(004)	(005)	(006)	(007)
I(calc.)	0.5	5.7	10.0	6.4	0.6	0.01	5.5
I(obs.)	$\frac{1}{2}$	$5\frac{1}{2}$	10.	6	1	vvw	5

To verify the chosen parameters, they were used to compute intensities of all the planes within the range of the powder pattern. The result is shown in Table 1 in which the calculated values are compared with those of the natural and artificial material. The agreement is unusually good and shows that the chosen parameters have been accurately determined.

TABLE 2. RICKARDITE ( $\text{Cu}_{4-x}\text{Te}_2$ ) AND  $\text{Cu}_{4-x}\text{Sb}_2$ :  
COMPARISON OF STRUCTURAL DATA

$\text{Cu}_{4-x}\text{Te}_2$		$\text{Cu}_{4-x}\text{Sb}_2$	
Space-group	<i>P4/nmm</i>		<i>P4/nmm</i>
<i>a</i>	3.97		3.992
<i>c</i>	6.11		6.091
<i>z</i> <sub>1</sub>	0.27		0.27
<i>z</i> <sub>2</sub>	0.715		0.70
Cu in ( <i>a</i> ) to 4 Cu in ( <i>a</i> )	2.81	Cu in ( <i>a</i> ) to 4 Cu in ( <i>a</i> )	2.82
4 Cu in ( <i>c</i> )	2.58	4 Cu in ( <i>c</i> )	2.59
4 Te in ( <i>c</i> )	2.72	4 Sb in ( <i>c</i> )	2.70
Cu in ( <i>c</i> ) to 4 Cu in ( <i>a</i> )	2.58	Cu in ( <i>c</i> ) to 4 Cu in ( <i>a</i> )	2.59
4 Te in ( <i>c</i> )	2.81	4 Sb in ( <i>c</i> )	2.83
1 Te in ( <i>c</i> )	2.64	1 Sb in ( <i>c</i> )	2.62
Te in ( <i>c</i> ) to 4 Cu in ( <i>a</i> )	2.72	Sb in ( <i>c</i> ) to 4 Cu in ( <i>a</i> )	2.70
4 Cu in ( <i>c</i> )	2.81	4 Cu in ( <i>c</i> )	2.83
1 Cu in ( <i>c</i> )	2.64	1 Cu in ( <i>c</i> )	2.62

The structure of rickardite is illustrated in Fig. 4 in which the fully occupied Cu positions are shown by solid black circles, the statistically half-occupied Cu positions, by the small blank circles, and the Te positions by the larger blank circles. The shortest atomic connections are shown in the figure and their lengths are listed in Table 2 which gives a full comparison of the structural data of rickardite and the corresponding antimonide of copper. It will be seen that there is very little difference between the two sets of interatomic distances. The corresponding Cu-Cu distances differ by only 0.01 and the Cu-Te distances are 0.02 greater than the corresponding Cu-Sb distances. This close similarity is in keep-

ing with the fact that Te immediately follows Sb in atomic number. As shown in Fig. 4, the structure of rickardite and the corresponding Sb compound is a layer structure in which the close packed layers are relatively weakly connected by the vertical bonds joining the roughly half-filled Cu positions to the Te atoms. This is in keeping with the basal cleavage which is common to both these compounds.

From the close similarity of the structures of rickardite and copper antimonide, it was to be expected that the powder patterns of the two compounds would be nearly alike. In attempting to prepare the copper antimonide, it was discovered that the antimonide apparently also has a defective composition. A fusion with the composition  $\text{Cu}_3\text{Sb}$  gave an intergrowth of the compound previously considered to be pure  $\text{Cu}_2\text{Sb}$ , together with a substantial amount of a second phase. On the other hand, a fusion with the composition  $\text{Cu}:\text{Sb}=4:3$  gave a purer product, corresponding to rickardite. It may be seen from the powder photographs (Figs. 1, 2, 3) that the powder pattern of this defective copper antimonide  $\text{Cu}_{2-x}\text{Sb}$  closely resembles those of natural and artificial rickardite. Our preparations of the antimonide showed the violet colour mentioned by Elander, Hägg & Westgren and explained by them as probably due to some polarization of the atoms; but this colour is not as arresting as the fiery purple-red of natural and artificial rickardite.

*Discussion.* Since the unit cell of rickardite and the identical artificial compound contains  $\text{Cu}_{4-x}\text{Te}_2$  with  $x \sim 1.2$  (30 per cent) the empirical formula  $\text{Cu}_4\text{Te}_3$  should be replaced by the cell formula; or, for simplicity, the composition could be written  $\text{Cu}_3\text{Te}_2$ , since  $x$  is fairly constant and near 1 in amount. Rickardite thus presents the unusual case of a defective structure with a large and almost invariant proportion of vacant metal positions. The ideal composition  $\text{Cu}_4\text{Te}_2$  is far beyond the existence range of the phase. It is true, the compound  $\text{Cu}_2\text{Te}$ , with only slight deficiency of Cu, is known in the artificial system and as the mineral weisite; but Thompson (1949) has confirmed the fact that this phase is entirely different from the rickardite phase. Thus rickardite differs from the familiar case of pyrrhotite,  $\text{Fe}_{2-x}\text{S}_2$ , in which  $x$  has a relatively small and continuous range, 0–0.4 (20 per cent); nor can rickardite be compared to the series  $\text{Ni}_{2-x}\text{Te}_2$ , in which  $x$  ranges continuously from 0 to 1 (50 per cent), giving  $\text{Ni}_2\text{Te}_2$  (B8-type, NiAs) to  $\text{NiTe}_2$  (melonite, C6-type,  $\text{CdI}_2$ ) (Klemm & Fratini, 1943).

The defective composition of rickardite indicates presence of Cu in two valence states,  $\text{Cu}^{\text{I}}$  and  $\text{Cu}^{\text{II}}$ . The simplified formula  $\text{Cu}_3\text{Te}_2$  can thus be written  $\text{Cu}_2\text{Te} \cdot \text{CuTe}$ , and from this it may be inferred that  $\text{Cu}^{\text{I}}$  fills the (a) positions while  $\text{Cu}^{\text{II}}$  partially fills the (c) positions. Presumably the empirical composition of umangite,  $\text{Cu}_3\text{Se}_2$ , which is physically similar to

rickardite but not isostructural, similarly represents  $\text{Cu}_2\text{Se} \cdot \text{CuSe}$ . These compounds recall the defective sulphide, digenite ("cubic chalcocite" or "blue chalcocite") with the cell formula  $\text{Cu}_{8-x}\text{S}_4$ . Typical examples closely conform to  $x=1$ , giving  $\text{Cu}_7\text{S}_4$  or  $3\text{Cu}_2\text{S} \cdot \text{CuS}$ , representing a sulphide of copper of two kinds, as originally recognized by Breithaupt (1844, in Dana, 1944, p. 181) and expressed by his name digenite.

Finally, a word on the remarkable blue colour of rickardite and other copper ores is in order. Is this connected with defective structure, or with the presence of Cu in two valence states, or is it due essentially to  $\text{Cu}^{\text{II}}$ ? The copper ore minerals whose body colours are blue in various degrees and the following:

Covellite, $\text{CuS}$ .....	indigo
Germanite, $\text{Cu}_3(\text{Fe, Ge})\text{S}_4$ or $3\text{CuS} \cdot (\text{Fe, Ge})\text{S}$ .....	purple
Rickardite, $\text{Cu}_3\text{Te}_2$ or $\text{Cu}_2\text{Te} \cdot \text{CuTe}$ .....	purple
Umanigite, $\text{Cu}_3\text{Se}_2$ or $\text{Cu}_2\text{Se} \cdot \text{CuSe}$ .....	purple
Digenite, $\text{Cu}_7\text{S}_4$ or $3\text{Cu}_2\text{S} \cdot \text{CuS}$ .....	blue

From these examples it would appear that the blue colour is due to copper atoms in the divalent state. The compound  $\text{Na}_{1-x}\text{WO}_3$  affords an analogous example of blue colour associated with valence greater than normal (Hägg, 1935). As the composition of this substance varies from  $x=0$  to  $x=\frac{2}{3}$  an increasing proportion of W ions are raised in valence from  $\text{W}^{+5}$  to  $\text{W}^{+6}$  and the colour changes progressively from yellow, through red, to blue.

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# CANCRINITE FROM BLUE MOUNTAIN, ONTARIO

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During the summer of 1947 while visiting the quarries of the American Nepheline Corporation at Blue Mountain in Methuen township, Ontario, Phoenix collected several specimens of fine, amber-yellow cancrinite. He subsequently analyzed selected material from the specimens on his return to Toronto. A portion was handed to Nuffield for further study. Professor M. A. Peacock and Mr. E. G. Robinson kindly undertook to measure the indices of refraction in Na light. Although chemical analyses of cancrinite with optical data are fairly plentiful in the literature, only one analysis is accompanied by cell dimensions. Therefore it was felt worthwhile to record the data collected during the study. This paper also discusses the chemical constitution of cancrinite and certain relationships between composition and optical properties.

The Blue Mountain cancrinite yielded a fragment showing the excellent hexagonal prismatic cleavage, permitting good rotation and Weissenberg films about the *c*-axis; these gave the cell dimensions<sup>3</sup> compared below with previous determinations:

<i>a</i>	<i>c</i>	
12.73	5.10	Monte Somma, Vesuvius (Zambonini & Ferrari, 1930)
12.60	5.18	Miask, Urals (Gossner & Mussnug, 1930)
12.72	5.18	Dódó, Korea (Kôzu & Takané, 1933)
12.60	5.12 kX	Blue Mountain, Methuen, Ontario (E. W. N.)

Two determinations of the specific gravity were made: 2.420 (R. P., on a piece weighing several grams), 2.423 (E. W. N., on a fragment (16 mg.) using the Berman microbalance).

Bragg (1937, p. 272), Berman (1937, pp. 365, 369), and others have concluded that cancrinite has a 3-dimensional framework of (Si,Al)-O<sub>4</sub> tetrahedra (silica type) with the ideal composition (Si<sub>6</sub>Al<sub>6</sub>O<sub>24</sub>)<sup>6-</sup>. Berman pictures this structural type as having large open spaces, some of which are occupied by CO<sub>3</sub> and SO<sub>4</sub> groups. Large cations enter the structure and neutralize the charge on these groups and on the framework. Specific gravities are characteristically low due to the open nature of the atomic structure. This view of cancrinite is supported by the data presented in Table 1 which gives the new analysis and cell contents calculated from the analysis, the measured specific gravity and the mass factor 1.650. Si and Al are both near 6, oxygen is near 24.

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<sup>3</sup> Using  $\lambda$  CuK $\alpha_1$  = 1.5374 kX.

When cancrinite analyses are compared they are usually recalculated to give atomic proportions on a basis of  $\text{Si} + \text{Al} = 12$  (Berman, 1937; Stewart, 1941). This almost invariably results in oxygen less than 24. It appears unlikely that water in the form of (OH) is substituting for oxygen. Zambonini & Ferrari (1930) found that the water is driven off at  $375^\circ \text{C}$ . without affecting the optical properties and concluded that it is not essential. Oxygen atoms are usually the largest in a silicate structure and they play an important part in determining the size and shape of the skeleton. It follows that the number of these atoms in a silicate formula is more apt to be constant than that of any other element. For this reason we have recalculated selected analyses of cancrinite for which optical

TABLE 1. ANALYSIS AND CELL CONTENTS OF CANCRINITE,  
BLUE MOUNTAIN, ONTARIO  
Analyst: R. Phoenix

$\text{SiO}_2$	33.98	Si	5.83
$\text{Al}_2\text{O}_3$	29.11	Al	5.88
$\text{Na}_2\text{O}$	18.69	Na	6.22
$\text{K}_2\text{O}$	0.64	K	0.14
$\text{CaO}$	4.80	Ca	0.88
$\text{CO}_2$	7.00	$\text{CO}_3$	1.64
$\text{SO}_3$	1.37	$\text{SO}_4$	0.18
Cl	0.42	Cl	0.12
$\text{H}_2\text{O}+$	4.34	$\text{H}_2\text{O}+$	2.48
$\text{H}_2\text{O}-$	0.23	O	22.66
O for $\text{Cl}_2$	-0.10		
100.48 <sup>4</sup>			

<sup>4</sup> Incl. FeO none, MgO none.

data are available on a basis of  $\text{O} = 24$  (Table 2). In all the analyses except 3 the sum  $\text{Si} + \text{Al}$  is now greater than 12. This shows that in cancrinite, as in other silicates, some Al substitutes for the cations.

Berman (1937) noted a tendency in the "normal" (i.e. nonsulphatic) cancrinites for Ca to increase as  $\text{Na} + \text{K}$  decreases. This is seen to be true also for sulphatic cancrinite, indicating isomorphous substitution of Ca for  $\text{Na} + \text{K}$ . Berman also observed a tendency for  $\text{CO}_3$  to increase with Ca. Similarly our table indicates an increase in  $\text{SO}_4$ , and consequent decrease in  $\text{CO}_3$ , with Na. It may be said that in general those natural cancrinites which are high in Ca are carbonic, while the cancrinites high in Na are sulphatic.

From the data in Table 2 the general chemical formula of cancrinite along with the maximum charges on the different groups of atoms may be

written as  $(\text{Na}, \text{K}, \text{Ca}, \text{Al})_{6-8}^{10+} \text{Si}_6 \text{Al}_6 \text{O}_{24}^{6-} \cdot (\text{SO}_4, \text{CO}_3)_{1-2}^{4-} \cdot 1-5 \text{H}_2\text{O}$ . This is in agreement with the formula of Berman (1937, p. 356), except that our formula shows inclusion of some Al with the cations and a greater range of water-content. The table indicates that the relation  $\text{Si}=\text{Al}=6$  is nearly realized in all analyses; the charge on the framework is therefore always close to 6-. However, the  $\text{CO}_3+\text{SO}_4$  content is usually less than 2 and may be as low as 1. Consequently the charge on these groups varies

TABLE 2. CANCRINITE: ATOMIC PROPORTIONS (O=24), OPTICAL PROPERTIES, AND SPECIFIC GRAVITIES

	1	2	3	4	5	6	7	8
Si.....	6.28	5.94	5.97	6.17	5.99	5.94	6.25	6.21
Al.....	5.94	6.21	5.96	6.23	6.16	6.21	6.01	6.06
Na.....	3.38	5.27	5.97	6.59	6.38	6.26	5.37	5.40
K.....	0.28	0.11	0.04	0.15	0.33	0.28	0.94	1.12
Ca.....	2.22	1.63	1.48	0.93	0.80	0.71	0.28	0.24
CO <sub>3</sub> .....	1.50	1.64	1.36	1.74	0.77	0.44	0.24	0.22
SO <sub>4</sub> .....	0.03	0.01	0.01	0.19	0.62	0.76	0.77	0.84
Cl.....	0.02	—	0.01	0.13	—	—	—	—
O.....	24.00	24.00	24.00	24.00	24.00	24.00	24.00	24.00
H <sub>2</sub> O.....	3.78	2.71	1.54	2.63	2.51	1.31	4.50	4.17
(Si, Al).....	12.00	12.00	11.93	12.00	12.00	12.00	12.00	12.00
(Na, K, Ca, Al)	6.10	7.16	7.49	8.07	7.66	7.40	6.85	7.03
(CO <sub>3</sub> , SO <sub>4</sub> , Cl) ..	1.55	1.65	1.38	2.06	1.39	1.20	1.01	1.06
ω.....	1.528	1.524	1.5238	1.515	1.509	1.502	1.492-1.493	
ε.....	1.503	1.501	1.5015	1.496	1.500	1.497		
ω-ε.....	0.025	0.023	0.0223	0.019	0.009	0.005	0.0007	
G.....	2.476	2.51	2.44	2.422	2.443	2.423	2.35	

1. Bancroft, Ont.; anal. Meen (1938). 2. Iron Hill, Gunnison Co., Colorado; anal. Larsen and Foshag (1926). Incl. Mg 0.10; Ti 0.01; Mn, Cl trace. 3. Dôdô, Korea; anal. Kôzu (1931). Incl. Mg 0.01; Fe''', P trace. 4. Blue Mountain, Methuen, Ontario; anal. Phoenix (this paper). Incl. Fe'', Mg none. Refractive indices by M. A. Peacock and E. G. Robinson. 5. Beaver Creek, Colorado; anal. Steiger (in Larsen and Steiger, 1916). Incl. Sr 0.01; Ti 0.01. 6. Allt á Mhuillinn, Loch Borolan, Assynt, Scotland; anal. Stewart (1941). Incl. Sr 0.03. 7. Ilmen Mts., Ural; anal. Morachevsky (in Zavaritzsky, 1929). Incl. Mg 0.03; Fe''' 0.03. 8. Ilmen Mts., Ural; anal. Morachevsky (in Zavaritzsky, 1929). Incl. Mg 0.03; Fe''' 0.01.

in different cancrinites and is characteristically less than 4-. This variation in negative charge together with considerable isomorphous substitution of Ca for Na results in a cation content which varies between 6 and 8. Therefore it may be assumed that the cancrinite structure practically always contains unfilled positions. This is borne out by the measured specific gravities of various cancrinites (Table 2). They are distinctly



lower than the value 2.59, calculated for  $\text{Na}_6\text{Ca}_2 \cdot \text{Al}_6\text{Si}_6\text{O}_{24} \cdot 2\text{CO}_3 \cdot 3\text{H}_2\text{O}$ , which presumably contains the maximum number of atoms. The available data do not point to any connection between the vacant positions and the water content.

Stewart (1941) noted that the optical properties are affected by variation in composition. He pointed out that with increasing  $\text{SO}_4$ ,  $\omega$  falls rapidly,  $\epsilon$  less so, resulting in a decrease in birefringence. The analyses in Table 2 have been arranged in order of decreasing birefringence caused by a decrease in  $\omega$  and somewhat irregular decrease in  $\epsilon$ . Although this is the order of increasing  $\text{SO}_4$  in the sulphatic cancrinites (4-8), it is also the order of decreasing Ca, and decreasing  $\text{Ca}/(\text{Na}+\text{K})$  for all the analyses. The indices of refraction and birefringence are characteristically higher in the calcic members of Ca-Na series such as the plagioclases, the scapolites, and some of the zeolites.

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## NOTES AND NEWS

### ANTIMONIAL SILVER ORE FROM COBALT, ONTARIO

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Qualitative examination of the specimen showed, besides silver and antimony, small quantities of bismuth with mercury. A quantitative analysis gave the following results, although the determination of the antimony was not wholly satisfactory:

Silver	84.10 per cent
Antimony	13.55
Bismuth	1.06
Mercury	0.52
Gangue	0.05
<hr/>	
	99.28

S, As, Se, Te, Au, Pt, Tl, Fe, Ca and similar metals could not be detected.

The silver is too high to correspond to the formula of "antimonide of silver,"  $\text{Ag}_3\text{Sb}$  ( $\text{Ag}=73.0$  per cent,  $\text{Sb}=27.0$  per cent). Although the formula  $\text{Ag}_7\text{Sb}$  might represent the composition, it seems unlikely that such a compound is to be assumed. The reasons for discarding a definite formula and considering the mineral to be an alloy of varying composition are as follows.

The composition seems to vary from point to point, as indicated by the inequality of the tarnishing in air. Mercury would undoubtedly diffuse with ease and so produce inequalities of composition. Moreover, alloys are not definite compounds as a rule. In this particular case alloys of silver and antimony over all ranges of composition have been thoroughly studied.<sup>2</sup> The melting points of both silver and antimony are lowered by

<sup>1</sup> Published by permission of the Director, U. S. Geological Survey. The MS of this note, dated April 15, 1910, was found among the papers left by the late Chief Chemist, R. C. Wells [1877-1944] and forwarded to me by Dr. Michael Fleischer and later again by Dr. Earl Ingerson, with permission to publish in *Contributions to Canadian Mineralogy*. The ore was collected by S. F. Emmons of the U. S. Geological Survey, and forwarded to C. W. Hayes (January 29, 1910) with a request that the Chief Chemist (then F. W. Clarke) should authorize its analysis. The analysis and discussion by R. C. Wells deserve publication as the first accurate description of the famous Cobalt silver, which has long been erroneously called dycrasite. In the light of recent work (*Univ. Toronto Studies, Geol. Ser.*, **44**, 31, 1940) the sample analyzed by Wells was probably an intergrowth of dycrasite ( $\text{Ag}_3\text{Sb}$ ) and antimonial silver ( $\text{Ag, Sb}$ ).—M.A.P.

<sup>2</sup> G. I. Petrenko, *Z. anorg. Chem.*, **50**, 139, 1906.

the presence of the other metal, reaching a eutectic at 45 per cent of antimony which melts at  $485^{\circ}$ . Alloys richer in antimony are found upon microscopic examination to consist of crystals of antimony and eutectic. Alloys containing 27 per cent of antimony are found to consist of one species of crystals which is in fact the compound  $\text{Ag}_3\text{Sb}$ , and this is the only compound which has been detected by the study of the artificial alloys. Alloys with small percentages of antimony consist of mixed crystals of silver and antimony, i.e. "solid solutions" of antimony in silver. The composition of this mineral falls in the last category. Its melting point, if the pure silver contained 13.5 per cent of antimony would be about  $790^{\circ}$ . As a matter of fact the presence of the bismuth would probably lower this point somewhat. Bismuth forms no definite compound with silver. The crystalline nature of the mineral is shown by the accompanying microphotograph (Fig. 1) of a surface which had been polished and etched with a mixture of nitric and tartaric acids. The method of lighting the specimen was not adapted to show the exact nature or form of the crystals.



FIG. 1

This mineral, therefore, consists of "mixed crystals," which are solid solutions of the metals of which it is composed, in each other.

With respect to the origin of such a deposit the simplest explanation seems to be that a mixture of metals or compounds has been reduced by organic matter, or hydrogen, but other explanations are by no means excluded.

FURTHER OCCURRENCES OF ANTIMONY AND TELLURIUM  
MINERALS IN WESTERN CANADA

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These notes identifying some of the less common ore minerals from Western Canada continue the series of notes previously published in *Univ. Toronto Studies*, Geol. Ser., **49**, 78, 1945; **50**, 75, 1946; **51**, 71, 1947; **52**, 82, 1948. The identifications have all been established or verified by *x*-ray powder photographs. Numerous students at the University of British Columbia assisted both in collecting and examining samples; the most deserving of mention are R. B. Campbell, L. B. Stark, and C. Cheriton. The writers are indebted to Professor M. A. Peacock for the use of the *x*-ray equipment in Toronto.

*Native bismuth and tellurbismuth.* Allan Lake, Yellowknife, N.W.T. At this property veins composed largely of quartz contain less than one per cent of metallic minerals, amongst which native bismuth is conspicuous. Arsenopyrite, chalcopyrite, sphalerite, gold, and tellurbismuth all occur in the vicinity of native bismuth.

*Berthierite.* Crestaurum Mine, Yellowknife, N.W.T. A single hand specimen of glassy quartz showed a 2 mm. area of a steel-gray compact fibrous mineral which proved to be berthierite.

*Jamesonite.* Morris Mine, Tatlayoko Lake, Clinton M.D., B.C. Arsenopyrite and stibnite, in veins consisting predominantly of quartz, are characteristic of this property. Sphalerite, tetrahedrite, galena, pyrite, native gold, and a carbonate, probably calcite, are all present in small amounts. Jamesonite may be found in quartz up to 1.5 mm., along fractures and disseminated as minute needles. The jamesonite is also intimately associated with both tetrahedrite and galena and less closely with stibnite; it occurs rarely veining sphalerite and arsenopyrite.

*Tetrahedrite.* Ajax Claim, Wavell Group, Clinton M.D., B.C. This claim situated at the head of the North Fork of Watson Bar Creek had previously produced minor amounts of placer gold but no vein had been discovered prior to 1948. In 1948 ground sluicing revealed a much weathered carbonate vein varying from a few inches to two feet in width, with scant sulphides of which tetrahedrite has so far been the only metallic mineral positively identified.

*Jamesonite.* Ten Mile Creek, Skagit River, Yale M.D., B.C. Through the kindness of Dr. H. Sargent, Chief Mining Engineer for the province of B.C., a specimen from this now deserted camp was obtained. Early reports referred to the presence of stibnite mixed with a number of sulphides, of which pyrrhotite and sphalerite were most abundant. No



stibnite could be found in the specimen but jamesonite was positively identified as the steel-gray mineral veining dark sphalerite.

*Boulangerite.* Mayflower Claim, Rossland Camp, Trail M.D. Through the kindness of Dr. M. Hedley of the B.C. Department of Mines a specimen of ore from this claim was obtained from Mr. S. Bruce. The ore consists of galena, sphalerite, pyrite, pyrrhotite, and arsenopyrite. Associated with these minerals was a fibrous one, in large compact areas up to 30 mm., whose identity was uncertain: it proved to be boulangerite.

*Petzite, Hessite, Altaite.* Sweetner Vein, Hedley Monarch Property, Olalla Camp, Osoyoos M.D., B.C. This vein varies in width from  $\frac{1}{4}$  inch to  $1\frac{1}{2}$  inches and is sparsely mineralized with pyrite, chalcopyrite, sphalerite, galena, tetrahedrite, gold, petzite, hessite, and altaite for 130 feet. The erratic distribution of the microscopic telluride minerals and gold leads to erratic gold and silver values. In view of the tendency of the tellurides to rapid decomposition, further careful prospecting in this area is suggested.

#### GOONGARRITE AND WARTHAITE DISCREDITED

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Goongarrite was first described by Simpson (*J. Roy. Soc. Western Australia*, **10**, 65, 1924) as a monoclinic lead bismuth sulphide from Lake Goongarrie, Comet Vale Township, Western Australia. It occurs as irregular to platy masses in part subfibrous and slightly radiating, in association with gold in a quartz vein in amphibolite. Analysis: Pb 54.26, Bi 28.81, S 15.24, rem. (Zn, Fe, Ag, Sb, Se) 1.63, total 99.94, giving the formula  $4\text{PbS} \cdot \text{Bi}_2\text{S}_3$ .

Dr. V. B. Meen of the Royal Ontario Museum kindly loaned a specimen labelled "goongarrite" (M14011) from the above locality. The specimen consists of a few silver-grey compact fibrous masses in white quartz. An x-ray powder photograph of these fibres yielded a complex pattern which proved to be that of a mixture of cosalite ( $2\text{PbS} \cdot \text{Bi}_2\text{S}_3$ ) and galena.

Warthaite is the name given by Krenner (*Mat. Termés Ért.*, **42**, 4, 1926) to a sulphide of bismuth and lead from Vaskö, Hungary. Analysis: Pb 54.53, Bi 28.18, S 15.31, rem. (Ag, Cu, Fe) 2.23, total 100.25.

Dr. Meen also loaned a specimen labelled "warthaite" (M13912) for study. It consists of white crystalline limestone with grains of sphalerite, pyrite, and hematite, also sparse acicular to radial aggregates of steely-grey "warthaite." An x-ray powder photograph of a radial aggregate gave a pattern identical with that given by "goongarrite." An x-ray photograph of the needles alone gave the cosalite pattern.

Dana (*Syst. Min.*, 1944, p. 402) suggests that "warthaite" is identical with "goongarrite" but that adequate data are lacking to establish the relationship. These observations confirm this suggestion and indicate that both are intimate mixtures of cosalite and galena.

#### LIQUID INCLUSIONS IN GEOTHERMOMETRY

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It was with considerable interest that I read the paper on "Liquid inclusions in halite as a guide to geologic thermometry" by Dreyer, Garrels and Howland in the January-February, 1949, issue of this Journal, but I am at variance with the interpretation put upon the results and the conclusions arrived at by the authors.

Some time ago a technique was developed in this department whereby the noise made by the breaking out (decrepitation) of liquid inclusions in minerals when heated, is used to indicate the point of complete filling by the liquid phase. This technique was described by Scott (*Econ. Geol.*, **43**, 637, 1948). Modifications have been made to the apparatus, particularly to the method of recording the onset of decrepitation, in an attempt to attain greater precision. In effect a curve is drawn, automatically, of the frequency of decrepitation against temperature. The inflection point in this curve is taken to indicate the temperature at which the fluid inclusions have just been completely filled by the liquid phase. This is described more fully elsewhere in this issue. In calibrating the apparatus and technique, synthetic mineral crystals grown under controlled conditions were used so far as possible, surprisingly close correspondence being obtained between the calculated temperatures of complete filling and those given by the apparatus. Among the minerals used was sodium chloride crystallized from a saturated aqueous solution at the temperature of the boiling point of the solution. Decrepitation gave a temperature in excess of this by  $0.5^{\circ}$  C. which is well within experimental error. Subsequently sodium chloride was grown from saturated aqueous solutions at temperature of  $45^{\circ}$  C.  $\pm 2^{\circ}$  C. and  $80^{\circ}$  C.  $\pm 2^{\circ}$  C. In both these cases decrepitation indicated temperatures of complete filling substantially the same as those of formation, certainly well within the possible experimental error.

In their paper, Dreyer, Garrels and Howland suggest that, since they obtained temperatures of formation of 70 to  $100^{\circ}$  C. for natural halite, using the direct observation-heating stage technique described by Inger-

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son (*Am. Mineral.*, **32**, 375, 1947), and since "it is obvious, that the bodies of water from which the sedimentary halite was deposited could not have had a general temperature of 70–100° C.," the use of liquid inclusions in geologic thermometry may not give a true indication of the general temperature of the solution from which the material was crystallised.

In view of the close correspondence between the temperature of the solution from which artificial halite was crystallised and the experimentally determined temperature of complete filling of the inclusions, as described above, an alternative conclusion to that of Dreyer, Garrels, and Howland is suggested.

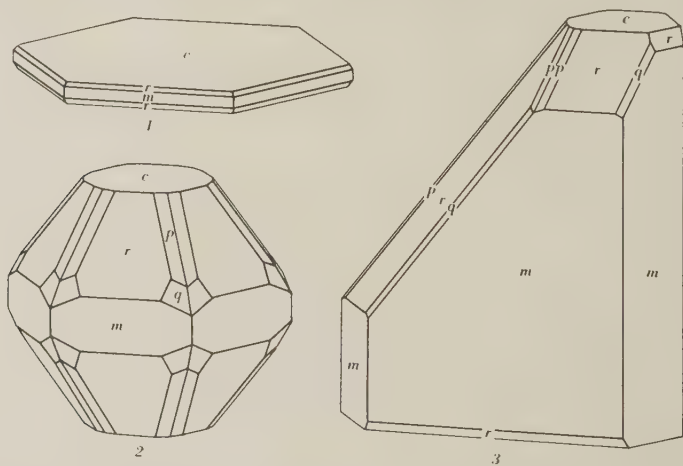
It is possible that the crystals examined were not those individual crystals initially precipitated from a subaerial body of water. The only evidence given that the halite had not suffered reworking was that the specimens came from dry, well stratified, undistorted strata. It is very unsafe however to assume that, because the beds show no distortion, recrystallisation has not taken place. In view of the high solubility of sodium chloride in water it would seem likely that recrystallisation, not only once but many times, would be the rule rather than the exception during the compaction of the original sediment. The connate water would be saturated with respect to sodium chloride and as the temperature rose with depth of burial it would tend to take more into solution. Nevertheless there must be a constant interchange of ions between the crystals and solution, the larger crystals growing at the expense of the small ones, material going into solution at points of high pressure and being precipitated at points of low pressure. Thus while the second of the criteria of primary inclusions, namely a good correlation between the volume of the inclusion and the volume of the gas phase, certainly indicates a primary nature for the inclusion with respect to the enclosing crystal, there is nothing to indicate that the crystal itself is primary to the deposit.

Therefore when using liquid inclusions to determine conditions of temperature and pressure of formation of a mineral, care must be exercised to examine the possibilities of recrystallisation subsequent to initial deposition, particularly where the mineral examined has a high solubility in water. If there is evidence that recrystallisation can have taken place, then the primary liquid inclusions can only indicate the temperature/pressure conditions prevailing during the last cycle of recrystallisation.

## ARTIFICIAL PYRRHOTITE

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Well-developed crystals of non-magnetic artificial pyrrhotite displaying two new crystallographic forms appeared in the products of certain experiments designed primarily for the synthesis of lead-bismuth sulphides from strongly alkaline sodium sulphide solutions. These solutions, held in a bomb at about 1200 bars and 400° C., attacked the steel walls and, after cooling, were found to have precipitated part of the iron in combination with the sulphur of the charges as lustrous bronze-brown crystals of pyrrhotite. Various habits, platy, equidimensional, columnar, and fibrous, were displayed by individuals sometimes reaching a greatest dimension of two or three millimetres. The perfection and excellent development of these crystals, as well as the rarity of good crystals of pyrrhotite in nature, made worthwhile a goniometric and x-ray study.



Portraits of typical habits are shown in Figs. 1 and 3; Fig. 2 shows ideal development of all the observed forms: the basal pinakoid  $c(0001)$ , the hexagonal prism  $m(10\bar{1}0)$ , the hexagonal dipyrmaid  $r(10\bar{1}1)$ , and the two new forms, dihexagonal dipyramids,  $p(21\bar{3}3)$ , and  $q(31\bar{4}3)$ . Several crystals are tabular on  $m$ , with dominant development of a zone of the type  $[(01\bar{1}1)(10\bar{1}0)]$ , large  $r$  faces and narrow  $p$  and  $q$  facets (Fig. 3). Ordinarily, only one termination is well-developed; the other usually displays a large basal pinkaoid modified by one or two narrow pyramidal faces, suggesting the hemimorphic class  $6mm$ .

<sup>1</sup> Research Council of Ontario Scholar, Department of Geological Sciences.



Excellent signals were obtained on the reflecting goniometer from  $c$ ,  $m$ , and  $r$ , permitting the calculation of an accurate morphological axial ratio

$$a':c'=1:1.6918$$

The signals from  $p$  and  $q$  were weak and blurred. Table 1 gives the observed and calculated angles from the fourteen crystals examined.

TABLE 1. ARTIFICIAL PYRRHOTITE. TWO-CIRCLE ANGLES

$$a':c'=1:1.6918; p_0:r_0=1.9535:1$$

Form	No. of Faces	Observed Range		Observed Mean		Calculated	
		$\phi$	$\rho$	$\phi$	$\rho$	$\phi$	$\rho$
$m$ (10 $\bar{1}0$ )	49	29° 40'–30° 20'	—	30° 00'	90° 00'	30° 00'	90° 00'
$r$ (10 $\bar{1}1$ )	40	29 56–30 02	62° 35'–63° 04'	30 00	62 53½	30 00	62 53½
$p$ (21 $\bar{3}3$ )	16	11 55–13 56	59 29–60 07	12 57	59 55	10 53½	59 50½
$q$ (31 $\bar{4}3$ )	8	15 43–18 15	66 31–66 58	17 32	66 40	16 06	66 56

Laue photographs with the beam perpendicular to the  $c$ -axis showed no lack of reflection symmetry across (0001), and indeed none could be expected unless pyrrhotite should afford an exception to Friedel's law, like zinckenite (Nuffield, 1946).

Rotation and Weissenberg films about the  $c$ - and  $a$ -axes showed the symmetry of the space group  $C6/mmc$ , and strong diffractions defining a pseudo-cell

$$a'=3.435, c'=5.811 \text{ kX};^2 c'/a'=1.692$$

Weak layer lines on the rotation films about the  $a$ - and  $c$ -axes gave evidence of a superstructure with  $a=3a'$ ,  $c=2c'$ . On the gnomonic projection of the forms, a partial influence of the superstructure on the morphology is shown by the position of the  $p$  and  $q$  face-nodes thirding the  $p_0$  dimension, corresponding to tripling of the  $a$  axis.

Representative recent measurements of the cell dimensions of artificial and natural crystals of  $\text{Fe}_{2-x}\text{S}_2$  are collected in Table 2. All the examples, except the pyrrhotite from Kuså, are hexagonal, with the small cell  $a'c'$  containing  $\text{Fe}_{2-x}\text{S}_2$ , and in some cases hexagonal superstructures  $ac$ . The data for Kuså fairly represent ten sets of similar values for monoclinic (nearly orthohexagonal) pyrrhotite from Sweden. It will be seen that  $a'$  is nearly constant while  $c'$ , and hence  $c'/a'$ , decreases with increasing shortage of Fe. Thus the new artificial crystals are apparently only slightly deficient in Fe. However the marked shrinkage of  $c'$  from the hexagonal to the monoclinic types with similar composition, appears to

<sup>2</sup> Using  $\text{CuK}\alpha_1$ ,  $\lambda=1.5374 \text{ kX}$ .

TABLE 2. CELL DIMENSIONS<sup>3</sup> OF MEMBERS OF THE SERIES  $\text{Fe}_{2-x}\text{S}_2$ 

		$a'$	$c'$	$c'/a'$	$a$	$c$	
Artificial	$\text{Fe}_{2.00}\text{S}_2$	3.433	5.860	1.707	$\sqrt{3}a'$	$2c'$	(1)
Artificial	—	3.435	5.811	1.692	$3a'$	$2c'$	(2)
Artificial	$\text{Fe}_{1.83}\text{S}_2$	3.437	5.746	1.672	$a'$	$c'$	(3)
Boliden	$\text{Fe}_{1.86}\text{S}_2$	3.437	5.722	1.665	$a'$	$c'$	(4)
Lavergruvan	$\text{Fe}_{1.76}\text{S}_2$	3.434	5.714	1.664	$a'$	$c'$	(4)
Kuså	$\text{Fe}_{1.82}\text{S}_2$	$b$ 3.428	$c$ 5.677	$c/b$ 1.656	$a$ 5.937	$\beta$ 89.60°	(4)
Schneeberg } Morro Velho }	—	3.435	5.68	1.65	$2a'$	$4c'$	(5)

(1) Hägg & Sucksdorff (1933). (2) A. R. G. (3) Haraldsen (1937). (4) Byström (1945). (5) Buerger (1947).

be due to a transformation discussed by Byström (1945). There is no agreement in the dimensions of the superstructures noted on these materials.

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<sup>3</sup> The previous values are all given as Å, but they are presumably all in kX.

LATTICE SPACINGS FOR GLANCING ANGLES WITH  
IRON X-RADIATIONS. KAIMAN, *Bureau of Mines, Ottawa, Ontario*

$\theta^\circ$	5	6	7	8	9	10	11	12	13	14
0.00	11.084	9.2419	7.9268	6.9411	6.1753	5.5631	5.0628	4.6463	4.2944	3.9931
0.05	10.974	9.1656	7.8708	6.8984	6.1414	5.5358	5.0402	4.6273	4.2782	3.9792
0.10	10.867	9.0908	7.8157	6.8561	6.1080	5.5086	5.0178	4.6085	4.2622	3.9654
0.15	10.762	9.0172	7.7612	6.8143	6.0749	5.4818	4.9955	4.5899	4.2463	3.9517
0.20	10.659	8.9448	7.7076	6.7730	6.0421	5.4552	4.9735	4.5713	4.2305	3.9380
0.25	10.557	8.8734	7.6547	6.7322	6.0098	5.4289	4.9517	4.5529	4.2147	3.9245
0.30	10.458	8.8034	7.6027	6.6919	5.9778	5.4028	4.9300	4.5347	4.1992	3.9110
0.35	10.361	8.7343	7.5513	6.6521	5.9461	5.3770	4.9086	4.5166	4.1838	3.8977
0.40	10.265	8.6664	7.5005	6.6129	5.9147	5.3514	4.8874	4.4987	4.1684	3.8845
0.45	10.171	8.5994	7.4504	6.5740	5.8837	5.3261	4.8663	4.4809	4.1533	3.8713
0.50	10.079	8.5336	7.4010	6.5357	5.8530	5.3010	4.8454	4.4632	4.1381	3.8582
0.55	9.9885	8.4688	7.3522	6.4977	5.8226	5.2762	4.8248	4.4458	4.1232	3.8453
0.60	9.8997	8.4048	7.3042	6.4603	5.7927	5.2516	4.8043	4.4284	4.1083	3.8324
0.65	9.8123	8.3420	7.2567	6.4232	5.7629	5.2272	4.7839	4.4112	4.0935	3.8196
0.70	9.7264	8.2800	7.2099	6.3865	5.7335	5.2031	4.7638	4.3941	4.0789	3.8069
0.75	9.6421	8.2188	7.1637	6.3502	5.7044	5.1792	4.7437	4.3771	4.0643	3.7943
0.80	9.5594	8.1587	7.1180	6.3145	5.6756	5.1554	4.7240	4.3603	4.0499	3.7817
0.85	9.4779	8.0995	7.0729	6.2791	5.6470	5.1319	4.7042	4.3437	4.0355	3.7693
0.90	9.3979	8.0410	7.0285	6.2441	5.6188	5.1087	4.6848	4.3271	4.0213	3.7569
0.95	9.3192	7.9834	6.9846	6.2095	5.5907	5.0857	4.6655	4.3107	4.0072	3.7446
$\theta^\circ$	15	16	17	18	19	20	21	22	23	24
0.00	3.7324	3.5047	3.3041	3.1262	2.9672	2.8245	2.6956	2.5788	2.4724	2.3751
0.05	3.7203	3.4941	3.2947	3.1177	2.9597	2.8177	2.6895	2.5732	2.4673	2.3704
0.10	3.7083	3.4835	3.2853	3.1094	2.9522	2.8110	2.6834	2.5677	2.4622	2.3658
0.15	3.6963	3.4730	3.2760	3.1011	2.9448	2.8043	2.6774	2.5622	2.4572	2.3612
0.20	3.6845	3.4626	3.2669	3.0929	2.9374	2.7977	2.6713	2.5567	2.4522	2.3566
0.25	3.6727	3.4522	3.2576	3.0848	2.9301	2.7911	2.6654	2.5512	2.4472	2.3521
0.30	3.6609	3.4419	3.2486	3.0766	2.9228	2.7845	2.6594	2.5458	2.4422	2.3475
0.35	3.6493	3.4317	3.2394	3.0685	2.9155	2.7779	2.6534	2.5404	2.4373	2.3430
0.40	3.6377	3.4215	3.2304	3.0605	2.9083	2.7714	2.6475	2.5350	2.4324	2.3385
0.45	3.6263	3.4114	3.2214	3.0525	2.9011	2.7649	2.6417	2.5297	2.4275	2.3340
0.50	3.6148	3.4013	3.2126	3.0445	2.8939	2.7584	2.6358	2.5244	2.4226	2.3295
0.55	3.6035	3.3913	3.2037	3.0365	2.8869	2.7520	2.6300	2.5191	2.4178	2.3250
0.60	3.5923	3.3814	3.1948	3.0287	2.8798	2.7456	2.6242	2.5137	2.4130	2.3206
0.65	3.5810	3.3716	3.1861	3.0209	2.8728	2.7393	2.6184	2.5085	2.4081	2.3162
0.70	3.5699	3.3617	3.1774	3.0131	2.8658	2.7329	2.6127	2.5033	2.4034	2.3118
0.75	3.5589	3.3520	3.1687	3.0053	2.8588	2.7267	2.6069	2.4980	2.3986	2.3074
0.80	3.5479	3.3423	3.1601	2.9976	2.8519	2.7204	2.6013	2.4929	2.3939	2.3031
0.85	3.5370	3.3327	3.1515	2.9899	2.8449	2.7141	2.5956	2.4877	2.3891	2.2987
0.90	3.5261	3.3231	3.1430	2.9824	2.8381	2.7079	2.5900	2.4826	2.3844	2.2944
0.95	3.5154	3.3136	3.1346	2.9747	2.8313	2.7018	2.5843	2.4775	2.3797	2.2901

$\theta^\circ$	25	26	27	28	29	30	31	32	33	34
0.00	2.2858	2.2037	2.1278	2.0577	1.9926	1.9321	1.8756	1.8230	1.7737	1.7275
0.05	2.2816	2.1997	2.1242	2.0543	1.9895	1.9291	1.8729	1.8204	1.7713	1.7253
0.10	2.2773	2.1958	2.1206	2.0510	1.9863	1.9262	1.8702	1.8179	1.7690	1.7231
0.15	2.2731	2.1919	2.1170	2.0476	1.9832	1.9234	1.8675	1.8154	1.7666	1.7209
0.20	2.2689	2.1880	2.1134	2.0443	1.9802	1.9204	1.8648	1.8128	1.7642	1.7187
0.25	2.2646	2.1841	2.1098	2.0410	1.9771	1.9176	1.8621	1.8103	1.7619	1.7164
0.30	2.2605	2.1803	2.1063	2.0377	1.9740	1.9147	1.8595	1.8078	1.7595	1.7143
0.35	2.2563	2.1765	2.1027	2.0344	1.9709	1.9119	1.8568	1.8053	1.7572	1.7121
0.40	2.2522	2.1727	2.0991	2.0311	1.9678	1.9090	1.8541	1.8029	1.7549	1.7099
0.45	2.2480	2.1688	2.0956	2.0278	1.9648	1.9062	1.8515	1.8004	1.7525	1.7077
0.50	2.2439	2.1650	2.0921	2.0246	1.9618	1.9034	1.8488	1.7979	1.7502	1.7055
0.55	2.2398	2.1612	2.0886	2.0213	1.9588	1.9006	1.8462	1.7955	1.7480	1.7034
0.60	2.2357	2.1575	2.0851	2.0180	1.9557	1.8978	1.8436	1.7930	1.7457	1.7012
0.65	2.2317	2.1537	2.0817	2.0148	1.9528	1.8950	1.8410	1.7906	1.7434	1.6991
0.70	2.2276	2.1500	2.0782	2.0116	1.9498	1.8922	1.8384	1.7881	1.7411	1.6969
0.75	2.2236	2.1462	2.0747	2.0084	1.9468	1.8894	1.8358	1.7857	1.7388	1.6948
0.80	2.2196	2.1425	2.0713	2.0053	1.9438	1.8866	1.8332	1.7833	1.7365	1.6927
0.85	2.2156	2.1388	2.0679	2.0021	1.9409	1.8839	1.8307	1.7809	1.7343	1.6906
0.90	2.2116	2.1352	2.0645	1.9989	1.9379	1.8811	1.8281	1.7785	1.7320	1.6884
0.95	2.2076	2.1315	2.0611	1.9957	1.9350	1.8784	1.8255	1.7761	1.7298	1.6863
$\theta^\circ$	35	36	37	38	39	40	41	42	43	44
0.00	1.6842	1.6435	1.6052	1.5691	1.5350	1.5029	1.4725	1.4437	1.4165	1.3907
0.05	1.6821	1.6415	1.6033	1.5673	1.5334	1.5013	1.4710	1.4423	1.4151	1.3894
0.10	1.6800	1.6396	1.6015	1.5656	1.5317	1.4998	1.4695	1.4409	1.4138	1.3882
0.15	1.6780	1.6376	1.5996	1.5639	1.5301	1.4982	1.4680	1.4395	1.4125	1.3869
0.20	1.6759	1.6356	1.5978	1.5621	1.5284	1.4966	1.4666	1.4381	1.4112	1.3856
0.25	1.6738	1.6337	1.5960	1.5604	1.5268	1.4951	1.4651	1.4367	1.4099	1.3844
0.30	1.6717	1.6318	1.5942	1.5587	1.5252	1.4936	1.4637	1.4354	1.4086	1.3832
0.35	1.6697	1.6298	1.5923	1.5569	1.5236	1.4920	1.4622	1.4340	1.4073	1.3819
0.40	1.6676	1.6279	1.5905	1.5552	1.5219	1.4905	1.4608	1.4326	1.4060	1.3807
0.45	1.6656	1.6260	1.5887	1.5535	1.5203	1.4890	1.4593	1.4313	1.4047	1.3795
0.50	1.6636	1.6241	1.5869	1.5518	1.5187	1.4875	1.4579	1.4299	1.4034	1.3783
0.55	1.6615	1.6221	1.5851	1.5501	1.5171	1.4859	1.4565	1.4285	1.4021	1.3770
0.60	1.6595	1.6202	1.5833	1.5484	1.5155	1.4844	1.4550	1.4272	1.4008	1.3758
0.65	1.6575	1.6183	1.5815	1.5467	1.5139	1.4829	1.4536	1.4258	1.3995	1.3746
0.70	1.6555	1.6164	1.5797	1.5450	1.5123	1.4814	1.4522	1.4245	1.3983	1.3734
0.75	1.6535	1.6145	1.5779	1.5434	1.5107	1.4799	1.4507	1.4231	1.3970	1.3722
0.80	1.6515	1.6127	1.5762	1.5417	1.5092	1.4784	1.4493	1.4218	1.3957	1.3710
0.85	1.6495	1.6108	1.5744	1.5400	1.5076	1.4769	1.4479	1.4205	1.3944	1.3698
0.90	1.6475	1.6089	1.5726	1.5384	1.5060	1.4754	1.4465	1.4191	1.3932	1.3685
0.95	1.6455	1.6071	1.5708	1.5367	1.5044	1.4739	1.4451	1.4178	1.3919	1.3674

The table of lattice spacings for glancing angles with copper x-radiation, by Forman (*Univ. Toronto Studies*, Geol. Ser., **51**, 87, 1947) has proved most convenient, especially as reprinted on a 4-page folder. The



$\theta^\circ$	45	46	47	48	49	50	51	52	53	54
0.00	1.3662	1.3430	1.3209	1.2999	1.2800	1.2611	1.2431	1.2259	1.2096	1.1941
0.05	1.3650	1.3418	1.3198	1.2989	1.2790	1.2601	1.2422	1.2251	1.2088	1.1933
0.10	1.3638	1.3407	1.3187	1.2979	1.2781	1.2592	1.2413	1.2242	1.2080	1.1926
0.15	1.3626	1.3396	1.3177	1.2969	1.2771	1.2583	1.2404	1.2234	1.2072	1.1918
0.20	1.3614	1.3384	1.3166	1.2959	1.2761	1.2574	1.2395	1.2226	1.2064	1.1911
0.25	1.3603	1.3373	1.3155	1.2949	1.2752	1.2565	1.2387	1.2217	1.2056	1.1903
0.30	1.3591	1.3362	1.3145	1.2938	1.2742	1.2556	1.2378	1.2209	1.2049	1.1896
0.35	1.3579	1.3351	1.3134	1.2928	1.2733	1.2546	1.2369	1.2201	1.2041	1.1888
0.40	1.3567	1.3340	1.3124	1.2918	1.2723	1.2537	1.2361	1.2193	1.2033	1.1881
0.45	1.3556	1.3329	1.3113	1.2908	1.2714	1.2529	1.2352	1.2185	1.2025	1.1873
0.50	1.3544	1.3318	1.3103	1.2898	1.2704	1.2519	1.2344	1.2176	1.2017	1.1866
0.55	1.3533	1.3307	1.3092	1.2888	1.2695	1.2510	1.2335	1.2168	1.2010	1.1859
0.60	1.3521	1.3296	1.3082	1.2878	1.2685	1.2501	1.2327	1.2160	1.2002	1.1851
0.65	1.3509	1.3285	1.3071	1.2869	1.2676	1.2493	1.2318	1.2152	1.1994	1.1844
0.70	1.3498	1.3274	1.3061	1.2859	1.2666	1.2484	1.2309	1.2144	1.1986	1.1837
0.75	1.3486	1.3263	1.3051	1.2849	1.2657	1.2475	1.2301	1.2136	1.1979	1.1829
0.80	1.3475	1.3252	1.3040	1.2839	1.2648	1.2466	1.2293	1.2128	1.1971	1.1822
0.85	1.3464	1.3241	1.3030	1.2829	1.2638	1.2457	1.2284	1.2120	1.1964	1.1815
0.90	1.3452	1.3230	1.3020	1.2819	1.2629	1.2448	1.2276	1.2112	1.1956	1.1808
0.95	1.3441	1.3220	1.3009	1.2810	1.2620	1.2439	1.2267	1.2104	1.1948	1.1800
$\theta^\circ$	55	56	57	58	59	60	61	62	63	64
0.00	1.1793	1.1653	1.1519	1.1391	1.1270	1.1155	1.1045	1.0941	1.0842	1.0748
0.05	1.1786	1.1646	1.1512	1.1385	1.1264	1.1149	1.1040	1.0936	1.0837	1.0744
0.10	1.1779	1.1639	1.1506	1.1379	1.1258	1.1143	1.1034	1.0931	1.0832	1.0739
0.15	1.1771	1.1632	1.1499	1.1373	1.1252	1.1138	1.1029	1.0926	1.0828	1.0734
0.20	1.1764	1.1625	1.1493	1.1367	1.1247	1.1132	1.1024	1.0921	1.0823	1.0730
0.25	1.1757	1.1618	1.1486	1.1360	1.1241	1.1127	1.1019	1.0916	1.0818	1.0725
0.30	1.1750	1.1612	1.1480	1.1354	1.1235	1.1121	1.1013	1.0911	1.0813	1.0721
0.35	1.1743	1.1605	1.1473	1.1348	1.1229	1.1116	1.1008	1.0906	1.0809	1.0716
0.40	1.1736	1.1598	1.1467	1.1342	1.1223	1.1110	1.1003	1.0901	1.0804	1.0712
0.45	1.1729	1.1591	1.1460	1.1336	1.1217	1.1105	1.0998	1.0896	1.0799	1.0707
0.50	1.1722	1.1585	1.1454	1.1330	1.1212	1.1099	1.0992	1.0891	1.0794	1.0703
0.55	1.1715	1.1578	1.1448	1.1324	1.1206	1.1094	1.0987	1.0886	1.0790	1.0698
0.60	1.1708	1.1571	1.1441	1.1318	1.1200	1.1088	1.0982	1.0881	1.0785	1.0694
0.65	1.1701	1.1565	1.1435	1.1312	1.1194	1.1083	1.0977	1.0876	1.0780	1.0690
0.70	1.1694	1.1558	1.1429	1.1306	1.1189	1.1077	1.0972	1.0871	1.0776	1.0685
0.75	1.1687	1.1552	1.1422	1.1300	1.1183	1.1072	1.0967	1.0866	1.0771	1.0681
0.80	1.1680	1.1545	1.1416	1.1294	1.1177	1.1066	1.0961	1.0861	1.0766	1.0676
0.85	1.1673	1.1538	1.1410	1.1288	1.1172	1.1061	1.0956	1.0857	1.0762	1.0672
0.90	1.1666	1.1532	1.1404	1.1282	1.1166	1.1056	1.0951	1.0852	1.0757	1.0668
0.95	1.1659	1.1525	1.1398	1.1276	1.1160	1.1050	1.0946	1.0847	1.0753	1.0663

present table for iron  $x$ -radiation, in the same form as Forman's table, is therefore offered for this commonly used radiation. Although the relation between Siegbahn's X.U. and true A.U. is now better known, there is

$\theta^\circ$	65	66	67	68	69	70	71	72	73	74
0.00	1.0659	1.0575	1.0494	1.0419	1.0348	1.0280	1.0217	1.0157	1.0102	1.0050
0.05	1.0655	1.0570	1.0491	1.0415	1.0344	1.0277	1.0214	1.0155	1.0099	1.0047
0.10	1.0650	1.0566	1.0487	1.0412	1.0341	1.0274	1.0211	1.0152	1.0096	1.0045
0.15	1.0646	1.0562	1.0483	1.0408	1.0337	1.0270	1.0208	1.0149	1.0094	1.0042
0.20	1.0642	1.0558	1.0479	1.0404	1.0334	1.0267	1.0205	1.0146	1.0091	1.0040
0.25	1.0638	1.0554	1.0475	1.0401	1.0330	1.0264	1.0202	1.0143	1.0088	1.0037
0.30	1.0633	1.0550	1.0472	1.0397	1.0327	1.0261	1.0199	1.0140	1.0086	1.0035
0.35	1.0629	1.0546	1.0468	1.0393	1.0324	1.0258	1.0196	1.0137	1.0083	1.0032
0.40	1.0625	1.0542	1.0464	1.0390	1.0320	1.0254	1.0193	1.0135	1.0080	1.0030
0.45	1.0620	1.0538	1.0460	1.0386	1.0317	1.0251	1.0190	1.0132	1.0078	1.0027
0.50	1.0616	1.0534	1.0456	1.0383	1.0313	1.0248	1.0187	1.0129	1.0075	1.0025
0.55	1.0612	1.0530	1.0452	1.0379	1.0310	1.0245	1.0184	1.0126	1.0073	1.0022
0.60	1.0608	1.0526	1.0449	1.0376	1.0307	1.0242	1.0181	1.0123	1.0070	1.0020
0.65	1.0604	1.0522	1.0445	1.0372	1.0303	1.0239	1.0178	1.0121	1.0067	1.0018
0.70	1.0599	1.0518	1.0441	1.0369	1.0300	1.0236	1.0175	1.0118	1.0065	1.0015
0.75	1.0595	1.0514	1.0437	1.0365	1.0297	1.0232	1.0172	1.0115	1.0062	1.0013
0.80	1.0591	1.0510	1.0434	1.0361	1.0293	1.0229	1.0169	1.0113	1.0060	1.0011
0.85	1.0587	1.0506	1.0430	1.0358	1.0290	1.0226	1.0166	1.0110	1.0057	1.0008
0.90	1.0583	1.0502	1.0426	1.0355	1.0287	1.0223	1.0163	1.0107	1.0055	1.0006
0.95	1.0579	1.0499	1.0423	1.0351	1.0283	1.0220	1.0160	1.0104	1.0052	1.0003
$\theta^\circ$	75	76	77	78	79	80	81	82	83	84
0.00	1.0001	0.9956	0.9914	0.9876	0.9841	0.9809	0.9781	0.9755	0.9733	0.9714
0.05	0.9999	0.9954	0.9912	0.9874	0.9839	0.9808	0.9779	0.9754	0.9732	0.9713
0.10	0.9996	0.9952	0.9910	0.9873	0.9838	0.9806	0.9778	0.9753	0.9731	0.9712
0.15	0.9994	0.9949	0.9909	0.9871	0.9836	0.9805	0.9777	0.9752	0.9730	0.9711
0.20	0.9992	0.9947	0.9906	0.9869	0.9834	0.9803	0.9775	0.9751	0.9729	0.9710
0.25	0.9989	0.9945	0.9904	0.9867	0.9833	0.9802	0.9774	0.9749	0.9728	0.9709
0.30	0.9987	0.9943	0.9903	0.9865	0.9831	0.9800	0.9773	0.9748	0.9727	0.9708
0.35	0.9985	0.9941	0.9901	0.9863	0.9830	0.9799	0.9771	0.9747	0.9726	0.9708
0.40	0.9983	0.9939	0.9899	0.9862	0.9828	0.9797	0.9770	0.9746	0.9725	0.9707
0.45	0.9980	0.9937	0.9897	0.9860	0.9826	0.9796	0.9769	0.9745	0.9724	0.9706
0.50	0.9978	0.9935	0.9895	0.9858	0.9825	0.9795	0.9768	0.9744	0.9723	0.9705
0.55	0.9976	0.9933	0.9893	0.9856	0.9823	0.9793	0.9766	0.9742	0.9722	0.9704
0.60	0.9974	0.9931	0.9891	0.9855	0.9822	0.9792	0.9765	0.9741	0.9721	0.9703
0.65	0.9971	0.9929	0.9889	0.9853	0.9820	0.9790	0.9764	0.9740	0.9720	0.9703
0.70	0.9969	0.9927	0.9887	0.9851	0.9819	0.9789	0.9762	0.9739	0.9719	0.9702
0.75	0.9967	0.9925	0.9885	0.9850	0.9817	0.9787	0.9761	0.9738	0.9718	0.9701
0.80	0.9965	0.9922	0.9883	0.9848	0.9815	0.9786	0.9760	0.9737	0.9717	0.9700
0.85	0.9963	0.9920	0.9882	0.9846	0.9814	0.9785	0.9759	0.9736	0.9716	0.9700
0.90	0.9960	0.9918	0.9880	0.9844	0.9812	0.9783	0.9758	0.9735	0.9715	0.9699
0.95	0.9958	0.9916	0.9878	0.9843	0.9811	0.9782	0.9756	0.9734	0.9714	0.9698

some advantage in retaining Siegbahn's scale. The table has therefore been computed for  $\lambda \text{FeK}\alpha_1 = 1.932076 \text{ kX}$ .

# WALKER MINERALOGICAL CLUB<sup>1</sup>

## BY-LAWS<sup>2</sup>

1. The Club shall be known as The Walker Mineralogical Club (Toronto, Canada).
2. The headquarters of the Club shall be the Royal Ontario Museum of Geology and Mineralogy, 100 Queen's Park, Toronto 5, Canada.
3. The object of the Club shall be to encourage scientific and popular mineralogy in Canada.
4. To promote scientific mineralogy, the Club shall offer annually a prize of one hundred dollars (\$100.00), to be known as the Walker Mineralogical Club Prize, for the best scientific paper on pure or applied mineralogy (including crystallography, petrology and geochemistry) submitted by a graduate student at any university or similar institution.

## MEMBERS

5. Ordinary membership is open to all interested persons and institutions.
6. The Council may elect Honorary Members from those who have rendered conspicuous service to mineralogy, and other eminent persons.

## COUNCIL

7. The Council of the Club shall consist of the following Officers: President, Past President, Secretary-Treasurer, Editor, Councillor for the Royal Ontario Museum of Geology and Mineralogy, and two Councillors-at-large. At least one of the officers shall be a Professor in the Department of Geological Sciences, University of Toronto.
8. The Council may elect an Honorary President, who shall also be a member of Council.
9. The members of Council, other than the Honorary President, shall be elected annually.
10. Council shall be authorized to fill vacancies in the Council during its term of office.
11. Only the President and the Secretary-Treasurer shall be authorized to draw cheques on the bank account of the Club for the payment of bills.

## MEETINGS

12. The meetings shall be held in the months of February, April, October, and December, on the second Thursday, or at more convenient dates specified by Council. These meetings are open to students and the general public.
13. Regular meetings may be combined with field excursions sponsored by the Club. Non-members (other than students) shall be charged registration fees, set by Council, for such excursions.

## PUBLICATION

14. The publication sponsored by the Club, *Contributions to Canadian Mineralogy*, appears as a special issue of *The American Mineralogist* (Journal of the Mineralogical Society of America). A copy of the publication shall be sent to each member in good standing.

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<sup>1</sup> Founded in 1938 and named in honour of the late Professor T. L. Walker (1867-1942), then Professor Emeritus of Mineralogy and Petrography in the University of Toronto and Director of the Royal Ontario Museum of Mineralogy.

<sup>2</sup> Proposed revision, 1949.



## PRIZE AND PUBLICATION FUND

15. Council shall maintain and administer a separate fund, to be known as the Prize and Publication Fund, to provide the Walker Mineralogical Club prize and to meet occasional special publication expenses.

## DUES

16. The annual membership fee, with the subscription to the publication, shall be \$2.50. Fees are payable on January 1. Members who are two years or more in arrears in payment shall be dropped from the membership list.

## AMENDMENTS

17. This constitution may be amended by a two-thirds majority of those voting on such amendments by means of a letter ballot sent to all members in good standing.

## OFFICERS, 1948

*Honorary President*, A. L. Parsons. *President*, G. E. Steel. *Past President*, F. Ebbutt. *Secretary-Treasurer*, W. M. Tovell. *Editor*, M. A. Peacock. *Councillors*, R. B. Ferguson, M. H. Froberg, A. R. Graham, V. B. Meen, L. Smith.

## ABSTRACT OF PROCEEDINGS, 1948

*February 10, 1948.* The President and Mrs. Steel graciously entertained the members of the club and wives at their home where Mr. Steel's fine mineral collection and lapidary equipment were on display.

*April 14, 1948.* After an introduction by Professor M. A. Peacock, the graduate students of the Department of Geological Sciences, University of Toronto, doing research in Mineralogy, presented summaries of their work during the academic year 1947-1948 as follows:

A. G. Brook: Crystal properties of some Werner complexes.

J. W. Earley: The PbSe-PbS system.

A. R. Graham: Crystals of artificial pyrrhotite.

I. H. Milne: Definitive description of chloritoid.

S. A. Forman: X-ray study of the mica group.

*October 15-17, 1948.* The Club met at Kingston, Ontario, for the first time, for the annual mineral collecting trip. Quartz crystals and a variety of pegmatite minerals were collected from the Precambrian rocks within a radius of fifty miles of Kingston. Dr. J. E. Hawley and Dr. L. G. Berry of the Department of Mineralogy, Queen's University, acted as leaders.

*December 9, 1948.* Mr. E. B. Tiffany, gemmologist with the firm of Henry Birks and Sons Ltd., Toronto, spoke to the Club on the subject "Man-made gems." His talk was illustrated by both synthetic and natural gem-stones. Mr. I. H. Milne received the Walker Club Prize for 1948, for his essay entitled "Chloritoid from Megantic County, Quebec" (p. 422).

WALTER M. TOVELL  
*Secretary-Treasurer*



# MALACHITE from THREE CONTINENTS

A short time ago we had an opportunity to secure an accumulation of MALACHITE that was originally from a lapidary who imported cutting material that met rigid specifications. The material had been purchased from Russian, South African, and American sources prior to 1915. The lot consisted only of cutting material of highest quality and it is a pleasure to offer this MALACHITE in specimens that every collector will be proud to own and exhibit.

## RUSSIAN MALACHITE

MALACHITE from Russian mines has been used for a great number of years for ornamental purposes since it has beautiful round bandings and various shades of green color. Polished slabs at  $1\frac{1}{2}" \times 2"$  \$2.00;  $2" \times 2"$  \$2.50;  $2" \times 3"$  \$3.00;  $2\frac{1}{2}" \times 3\frac{1}{2}"$  \$4.00;  $3" \times 4"$  \$5.00, \$6.00 and \$7.00 each.

Chunky pieces in the rough are available for carving, inlay work, sphere cutting or gem cutting. Pieces will run between two pounds and five pounds each at \$6.00 per pound.

## RHODESIAN MALACHITE

MALACHITE from South Africa is also prized for its compactness, beautiful bandings and is easy to work. Specimens we offer are mostly of straight bandings.  $1\frac{1}{2}" \times 2"$  \$1.50;  $1\frac{1}{2}" \times 2\frac{1}{2}"$  \$2.00;  $1\frac{1}{2}" \times 3"$  \$2.50;  $1\frac{1}{2}" \times 3\frac{1}{2}"$  \$3.00;  $1\frac{1}{2}" \times 4"$  \$3.50.

This is also available in unpolished slabs  $1\frac{1}{2}" \times 2"$  to  $1\frac{1}{2}" \times 4"$  and  $\frac{5}{16}"$  thick at 35¢ per square inch. Minimum order \$2.00.

## ARIZONA MALACHITE

MALACHITE from Arizona that is suitable for polishing is comparable to the finest Russian MALACHITE but of slightly darker shades of green. Selected polished specimens of rich color and bandings polished on rounded surfaces  $1" \times 1\frac{1}{2}"$  \$1.00;  $1" \times 2"$  \$1.50;  $2" \times 2"$  \$2.00;  $2" \times 3"$  \$2.50 and \$3.00; larger pieces at \$5.00 and \$7.50 each.

*Postage and insurance charges are additional.*

*Any overpayment will be returned at once.*

## SCHORTMANN'S MINERALS

6 MCKINLEY AVENUE

EASTHAMPTON, MASS.



## WARD'S *offers newly* *acquired* MINERALS *from* SWEDEN

Ward's is pleased to offer these choice mineral specimens from Sweden, plus other fine minerals from New Jersey and Canada.

### *from Sweden*

The four species listed below are selected from a large shipment of rare and interesting minerals from well-known mineral localities in Norway and Sweden. Complete and detailed listings of these minerals appear in Ward's Natural Science Bulletins, Vol. XXII, Nos. 4 and 5, March and May 1949. Write for a free copy.

**FERGUSONITE.** Ytterby, Sweden. A columbate-tantalate of yttrium, cerium, uranium, etc. The fergusonite occurs as dark brown masses in pegmatite. Sizes: average 2 x 2" to 3 x 4" and priced at \$3.50, \$4.00, \$5.00, \$6.00, \$7.50.

**KNEBELITE.** Alnö, Sweden. This iron manganese silicate is available in rich brownish black masses ranging from 2 x 2" to 2 x 3", priced at \$3.50, \$4.00, \$4.50, \$5.00; 2½ x 3" to 3½ x 3½" priced at \$6.00, \$7.50, \$8.00.

**RICHTERITE.** Langban, Sweden. An alkali manganese amphibole. Specimens consist of xld and xline brown masses in rock. Specimens average in size 2 x 2" to 2 x 3", priced at \$2.50, \$3.00, \$3.50; 2½ x 3½" to 3 x 4", priced at \$4.00, \$4.50, \$5.00.

**TELLUROBISMUTHITE.** Manganfallberget, Boliden, Sweden. This bismuth telluride occurs as rich xline masses in rock. Specimens average 1½ x 2" to 2 x 3", priced at \$4.00, \$5.00, \$6.00, \$7.50.

### *from Franklin, New Jersey*

**CHALCOPHANITE.** A hydrous oxide of zinc and manganese occurring as small bluish-black tabular crystals with hetaerolite. 2 x 3", priced at \$7.50; 2 x 3½", \$9.00; 3 x 5½", \$10.00.

**AXINITE.** A complex acid borosilicate of calcium, aluminum, and manganese with some iron and zinc. Xline yellow masses with polyadelphite, and/or fowlerite, biotite, etc. Sizes average 3 x 3", \$4.00, \$4.50, \$5.00; 4 x 4½" priced at \$7.50.

**WILLEMITE, var. Troostite.** Exceptionally large crystals with calcite and franklinite. 5 x 5½" priced at \$20.00.

**WILLEMITE (Phosphorescent).** The following specimens contain radiating masses of white willemite which is highly phosphorescent under short wave ultra violet light. Specimens average 1½ x 2" to 3 x 3" and are priced at \$2.00, \$2.50, \$3.00, \$4.00, \$5.00, \$6.00.

### *from Ontario, Canada*

**CYRTOLITE, near Bancroft, Ontario, Canada.** An altered zircon containing hafnium, uranium, etc. Sharp crystals and crystal groups average ½ to 1", priced at \$.35, \$.50, \$.75; 1 to 1¼", priced at \$1.00, \$1.50; 1½ to 2" are priced at \$2.00, \$2.50, \$3.00, \$3.50, \$4.00.

*All Prices Are List Price, Rochester, N.Y.*

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